

(FILE 'HOME' ENTERED AT 10:40:00 ON 18 SEP 2008)

L1 FILE 'REGISTRY' ENTERED AT 10:40:21 ON 18 SEP 2008
80 S (2-7)/LI AND P AND 3.5-8/O

L2 FILE 'CAPLUS' ENTERED AT 10:40:52 ON 18 SEP 2008
59 S L1
L3 8 S L2 AND (TI OR V OR CR OR MN)
L4 11 S L2 AND (FE OR CO OR NI OR CU OR ZR)
L5 6 S L2 AND (NB OR MO OR RU OR AG OR TA OR W OR PT OR AU)

=> s l3 or l4 or l5
L6 18 L3 OR L4 OR L5

=> d l-18 ibib ti it abs

L6 ANSWER 1 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2006:496431 CAPLUS <<LOGINID:20080918>>
DOCUMENT NUMBER: 145:145864
TITLE: A bimetallic, coordinated-ketene complex formed from a
bimetallic lithium-carbon spirocycle by
lithium-mediated insertion of CO into a
rhodium-carbon bond
AUTHOR(S): Fang, Min; Jones, Nathan D.; Lukowski, Robert;
Tjathas, Jim; Ferguson, Michael J.; Cavell, Ronald G.
CORPORATE SOURCE: Department of Chemistry, University of Alberta,
Edmonton, AB, T6G 2G2, Can.
SOURCE: Angewandte Chemie, International Edition (2006),
45(19), 3097-3101
CODEN: ACIEF5; ISSN: 1433-7851
PUBLISHER: Wiley-VCH Verlag GmbH & Co. KGaA
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 145:145864
TI A bimetallic, coordinated-ketene complex formed from a bimetallic
lithium-carbon spirocycle by lithium-mediated insertion of CO
into a rhodium-carbon bond
IT Insertion reaction
(bimetallic, ketene complex formed from bimetallic lithium-carbon
spirocycle by Li-mediated insertion of CO into rhodium-carbon
bond and crystal structures of reactant and product)
IT Carbene complexes
Spiro compounds
RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP
(Preparation); RACT (Reactant or reagent)
(bimetallic, ketene complex formed from bimetallic lithium-carbon
spirocycle by Li-mediated insertion of CO into rhodium-carbon
bond and crystal structures of reactant and product)
IT Alkenes, preparation
RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP
(Preparation); RACT (Reactant or reagent)
(heterocumulenes, ketenes, Li-Rh tetranuclear complex; bimetallic,
ketene complex formed from bimetallic lithium-carbon spirocycle by
Li-mediated insertion of CO into rhodium-carbon bond and
crystal structures of reactant and product)
IT Crystal structure
Molecular structure
(of lithium rhodium diene iminophosphoranylmethylene dinuclear and
carbonyl ketene tetranuclear complexes)
IT 12092-47-6, Bis[chloro(1,5-cyclooctadiene)rhodium] 230969-40-1

RL: RCT (Reactant); RACT (Reactant or reagent)
 (bimetallic, ketene complex formed from bimetallic lithium-carbon
 spirocycle by Li-mediated insertion of CO into rhodium-carbon
 bond and crystal structures of reactant and product)

IT 898550-45-3P
 RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP
 (Preparation); RACT (Reactant or reagent)
 (crystal structure; bimetallic, ketene complex formed from bimetallic
 lithium-carbon spirocycle by Li-mediated insertion of CO into
 rhodium-carbon bond and crystal structures of reactant and product)

IT 898550-48-6P
 RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
 (crystal structure; bimetallic, ketene complex formed from bimetallic
 lithium-carbon spirocycle by Li-mediated insertion of CO into
 rhodium-carbon bond and crystal structures of reactant and product)

IT 898550-47-5P
 RL: SPN (Synthetic preparation); PREP (Preparation)
 (hydrolysis product; bimetallic, ketene complex formed from bimetallic
 lithium-carbon spirocycle by Li-mediated insertion of CO into
 rhodium-carbon bond and crystal structures of reactant and product)

IT 898550-46-4P
 RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP
 (Preparation); RACT (Reactant or reagent)
 (mol. structure, hydrolysis; bimetallic, ketene complex formed from
 bimetallic lithium-carbon spirocycle by Li-mediated insertion of
 CO into rhodium-carbon bond and crystal structures of reactant
 and product)

AB A bimetallic spirocyclic bridged-carbene complex (1; carbene =
 (TMSN:PPh₂)₂C) was prepared and reacts almost quant. with CO by
 formal insertion into the Rh-C(Li) bond to form the dimeric tetranuclear
 complex 2, the 1st example of a μ_2, η^2 -(O,C) Li-Rh ketene complex.
 Complex 2 reacts with H₂O to transform the ketene ligand into a methanide
 group by CO elimination. The crystal and mol. structures of 1
 and 2·Et₂O were determined by x-ray crystallog.

REFERENCE COUNT: 60 THERE ARE 60 CITED REFERENCES AVAILABLE FOR THIS
 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 2 OF 18 CAPLUS COPYRIGHT 2008 ACS ON STN
 ACCESSION NUMBER: 2006:330088 CAPLUS <<LOGINID:20080918>>
 DOCUMENT NUMBER: 145:46176
 TITLE: Coordination chemistry of isomeric mixtures of linked
 di(phosphaguanidine) compounds: a spectroscopic and
 crystallographic study

AUTHOR(S): Mansfield, Natalie E.; Coles, Martyn P.; Avent,
 Anthony G.; Hitchcock, Peter B.
 CORPORATE SOURCE: Department of Chemistry, University of Sussex,
 Falmer/Brighton, BN1 9QJ, UK
 SOURCE: Organometallics (2006), 25(10), 2470-2474
 CODEN: ORGND7; ISSN: 0276-7333
 PUBLISHER: American Chemical Society
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 145:46176

TI Coordination chemistry of isomeric mixtures of linked di(phosphaguanidine)
 compounds: a spectroscopic and crystallographic study

IT Phosphines
 RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP
 (Preparation); RACT (Reactant or reagent)
 (di-; preparation and reactions of diastereomeric mixts. of rac- and
 meso-di(phosphaguanidine) compds. with aluminum and platinum to give
 bimetallic aluminum and monometallic platinum complexes)

IT Crystal structure
Molecular structure
(of diastereomeric di(phosphaguanidine) ligands and their bimetallic aluminum and monometallic platinum complexes)

IT 890054-94-1P 890055-36-4P
RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(crystal structure; preparation and reactions of diastereomeric mixts. of rac- and meso-di(phosphaguanidine) compds. with aluminum and platinum to give bimetallic aluminum and monometallic platinum complexes)

IT 889470-57-9P 889470-58-0P 889470-60-4P 889470-61-5P
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(crystal structure; preparation and reactions of diastereomeric mixts. of rac- and meso-di(phosphaguanidine) compds. with aluminum and platinum to give bimetallic aluminum and monometallic platinum complexes)

IT 75-24-1, Trimethylaluminum 538-75-0 554-68-7, Triethylammonium chloride 693-13-0 12080-32-9, Dichloro(1,5-cyclooctadiene)platinum 12266-72-7, (1,5-Cyclooctadiene)diiodoplatinum 125083-71-8
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation and reactions of diastereomeric mixts. of rac- and meso-di(phosphaguanidine) compds. with aluminum and platinum to give bimetallic aluminum and monometallic platinum complexes)

IT 889470-62-6P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and reactions of diastereomeric mixts. of rac- and meso-di(phosphaguanidine) compds. with aluminum and platinum to give bimetallic aluminum and monometallic platinum complexes)

IT 889470-59-1P 889470-63-7P 890055-48-8P 890055-50-2P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and reactions of diastereomeric mixts. of rac- and meso-di(phosphaguanidine) compds. with aluminum and platinum to give bimetallic aluminum and monometallic platinum complexes)

AB Multinuclear NMR spectroscopy and x-ray diffraction techniques were used to identify diastereomeric mixts. of rac- and meso-di(phosphaguanidine) compds. A brief survey of their coordination chemical demonstrated the formation of N,N'-bound bimetallic Al species and monometallic Pt complexes in which the ligand chelates to the metal through the two P donor atoms.

REFERENCE COUNT: 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 3 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2003:573904 CAPLUS <<LOGINID:20080918>>
DOCUMENT NUMBER: 140:62169
TITLE: Electrical conductivity in Li-Si-P-O-N oxynitride thin-films
AUTHOR(S): Lee, Seung-Joo; Bae, Jun-Hyun; Lee, Hee-Won; Baik, Hong-Koo; Lee, Sung-Man
CORPORATE SOURCE: Department of Metallurgical Engineering, Yonsei University, Seoul, 120-749, S. Korea
SOURCE: Journal of Power Sources (2003), 123(1), 61-64
CODEN: JPSODZ; ISSN: 0378-7753
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
TI Electrical conductivity in Li-Si-P-O-N oxynitride thin-films
IT Battery electrolytes
(elec. conductivity of Li-Si-P-O-N thin-film electrolytes for lithium batteries)
IT 639079-90-6 639079-91-7, Lithium nitride oxide phosphide

silicate (Li1.9NO0.26P(SiO3)0.28) 639079-92-8, Lithium nitride
oxide phosphide silicate (Li2.9N1.2600.1P(SiO4)0.35) 639079-93-9,
Lithium nitride oxide phosphide silicate (Li2.9N1.300.25P(SiO3)0.45)
RL: DEV (Device component use); PRP (Properties); USES (Uses)
(elec. conductivity of Li-Si-P-O-N thin-film electrolytes for lithium
batteries)

AB N-containing Li silicophosphate (LiSiPON) thin-film electrolytes, which
contain 2 glass-forming elements, are fabricated by sputtering from a
(1-x)Li3P04·xLi2SiO3 target in a N reactive plasma. The results of
impedance measurements show that the activation energy for conduction
decreases as the Si content increases, which increases the ionic conductivity
of
the films. These improvements in the elec. properties of the films are
due to the combined effect of the mixed former and N incorporation. The
decomposition potential of the electrolyte film in contact with Pt is
.apprx.5.5 V.

REFERENCE COUNT: 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 4 OF 18 CAPLUS COPYRIGHT 2008 ACS ON STN

ACCESSION NUMBER: 2003:364301 CAPLUS <<LOGINID:20080918>>

DOCUMENT NUMBER: 139:214572

TITLE: Reaction of a cyclopentadienylcobalt(II) phosphane
chelate with trimethylsilyl chloride and some
pseudohalides: unanticipated formation of new
cyclopentadienylcobalt(II) and (III) chelates

AUTHOR(S): Kakoschke, Alf; Yong, Li; Wartchow, Rudolf;
Butenschoen, Holger

CORPORATE SOURCE: Institut fuer Organische Chemie, Universitaet
Hannover, Hannover, D-30167, Germany

SOURCE: Journal of Organometallic Chemistry (2003), 674(1-2),
86-95

CODEN: JORCAI; ISSN: 0022-328X

PUBLISHER: Elsevier Science B.V.

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 139:214572

TI Reaction of a cyclopentadienylcobalt(II) phosphane chelate with
trimethylsilyl chloride and some pseudohalides: unanticipated formation of
new cyclopentadienylcobalt(II) and (III) chelates

IT Transition metal halides

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(cobalt, pseudohalides, crystal structure; preparation of
cyclopentadienylcobalt(II) (tethered phosphine) chloride and
pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT Sandwich compounds

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(half-sandwich, cobalt, tethered phosphine; preparation of
cyclopentadienylcobalt(II) (tethered phosphine) chloride and
pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT Oxidation

(halogenation; preparation of cyclopentadienylcobalt(II) (tethered
phosphine) chloride and pseudohalide complexes by trimethylhalosilane-
mediated halogenation)

IT Crystal structure

Molecular structure

(of cyclopentadienyl cobalt(II)-tethered phosphine chloride, isocyanate
and azide complexes)

IT Addition reaction, coordinative

(oxidative; of trimethylsilyl cyanide to give cobalt(III) dicyano
cyclopentadienyl (tethered phosphine) complex)

IT Halogenation
(preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 21502-53-4, Lithium diisopropylphosphide
RL: RCT (Reactant); RACT (Reactant or reagent)
(addition, oxidation; preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 176683-07-1P 588729-48-0P 588729-49-1P 588729-52-6P
588729-53-7P
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(crystal structure; preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 588729-51-5P
RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(mol. structure, lithiation, oxidation; preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 7677-24-9, Trimethylsilyl cyanide
RL: RGT (Reagent); RACT (Reactant or reagent)
(oxidative addition; preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 1118-02-1, Trimethylsilyl isocyanate 2290-65-5, Trimethylsilyl isothiocyanate
RL: RGT (Reagent); RACT (Reactant or reagent)
(oxidation reagent; preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 141847-75-8
RL: RCT (Reactant); RACT (Reactant or reagent)
(oxidation, halogenation; preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 1450-14-2P, Hexamethyldisilane
RL: BYP (Byproduct); PREP (Preparation)
(preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 588729-50-4P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

AB Reactions of the (η^2 -ethene)[$(\eta^5\eta^1-2-[(di-tert-butyl)phosphino]ethyl)cyclopentadienyl-\kappa P$]cobalt(I) chelate complex 1 with trimethylsilyl chloride and some pseudohalides were examined
Reaction of 1 with trimethylsilyl chloride, azide, isocyanate, and isothiocyanate gave cobalt(II) chelates [$(\eta^5\eta^1-C_5H_4CH_2CH_2PtBu_2-\kappa P)CoX$] [$2-5$, $X = Cl, N_3, NCO, NCS$], some of which were characterized by x-ray structure anal. In contrast to these reactions, treatment of 1 with trimethylsilyl cyanide resulted in the formation of cobalt(III) chelate [$(\eta^5\eta^1-C_5H_4CH_2CH_2PtBu_2-\kappa P)Co$ (CN) $_2$] (7). Reaction of 7 with $LiPr_2$ followed by oxidation gave a small yield of tetrametallic complex [$(\eta^5-tBuPOCH_2CH_2C_5H_4)Co$ (CN)($\mu-iPr_2POLi-\kappa P$) $_2$] (8), in which the two cobalt centers are bridged by an (LiO) $_2$ rhombus.

REFERENCE COUNT: 59 THERE ARE 59 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 5 OF 18 CAPLUS COPYRIGHT 2008 ACS ON STN
 ACCESSION NUMBER: 2002:894424 CAPLUS <<LOGINID::20080918>>
 DOCUMENT NUMBER: 138:106770
 TITLE: [Li{CH(Me)P(Ph)2(NCO2Me)}2(THF)2]: Crystal, Solution, and Calculated Structure of a N-Delocalized Lithium Phosphazene
 AUTHOR(S): Fernandez, Ignacio; Alvarez Gutierrez, Julia Maria; Kocher, Nikolaus; Leusser, Dirk; Stalke, Dietmar; Gonzalez, Javier; Ortiz, Fernando Lopez
 CORPORATE SOURCE: Area de Quimica Organica, Universidad de Almeria Carretera de Sacramento s/n, Almeria, 04120, Spain
 SOURCE: Journal of the American Chemical Society (2002), 124(51), 15184-15185
 CODEN: JACSAT; ISSN: 0002-7863
 PUBLISHER: American Chemical Society
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 138:106770
 TI [Li{CH(Me)P(Ph)2(NCO2Me)}2(THF)2]: Crystal, Solution, and Calculated Structure of a N-Delocalized Lithium Phosphazene
 IT Equilibrium
 (monomer-dimer; of alkyl di-Ph methoxycarbonyl phosphazene lithium complex)
 IT Crystal structure
 Molecular structure
 (of alkyl di-Ph methoxycarbonyl phosphazene lithium complex)
 IT MP2 (second-order Moller-Plesset method)
 (of delocalized lithium phosphazene complex)
 IT Molecular structure
 (optimized; of delocalized lithium phosphazene complex)
 IT 488082-70-8 488082-71-9
 RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)
 (preparation, crystal, solution, and calculated structure of a N-delocalized lithium phosphazene)
 IT 488082-73-1P
 RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
 (preparation, crystal, solution, and calculated structure of a N-delocalized lithium phosphazene)
 IT 127802-82-8
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (preparation, crystal, solution, and calculated structure of a N-delocalized lithium phosphazene)
 AB The first crystal structure of the lithium complex of a P-alkyl-P,P-diphenyl(N-methoxycarbonyl)phosphazene, [Li{CHMePPH2:NCO2Me}2(THF)2], prepared by treatment of CH3CH2P(Ph)2NCO2Me with BuLi in THF at -30° for 30 min and then layering the orange solution with hexane at -18° for 2 days, is described. It is dimeric, with the anion chelating the lithium in an unusual six-membered ring. A monomer-dimer equilibrium has been identified in THF solution Ab initio calcs.
 indicated that the six-membered ring is electronically favored over an alternative Li-C-P-N four-membered ring.
 REFERENCE COUNT: 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 6 OF 18 CAPLUS COPYRIGHT 2008 ACS ON STN
 ACCESSION NUMBER: 2002:84941 CAPLUS <<LOGINID::20080918>>
 DOCUMENT NUMBER: 136:279555
 TITLE: Water-Promoted Reaction of a Platinum(II) Oxo Complex with Ethylene

AUTHOR(S): Flint, Bruce; Li, Jian-Jun; Sharp, Paul R.
CORPORATE SOURCE: Department of Chemistry, University of Missouri-Columbia, Columbia, MO, 65211, USA
SOURCE: Organometallics (2002), 21(5), 997-1000
CODEN: ORGND7; ISSN: 0276-7333
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 136:279555
TI Water-Promoted Reaction of a Platinum(II) Oxo Complex with Ethylene
IT Rearrangement catalysts
(forming of platinum phosphine hydroxy complex as catalyst for oxygen atom transfer reaction in reaction of platinum oxo complexes with ethylene)
IT Crystal structure
Molecular structure
(of platinum lithium phosphine hydroxy bridged complex)
IT Substitution reaction, coordinative
(of platinum oxo complexes with ethylene, during which oxygen atom transfer takes place)
IT Rearrangement
(oxygen atom transfer; in reaction of platinum oxo complexes with ethylene, catalyzed by platinum phosphine hydroxy complex)
IT 406462-95-1P
RL: CAT (Catalyst use); SPN (Synthetic preparation); PREP (Preparation); USES (Uses)
(preparation and catalyst for oxygen atom transfer)
IT 406462-91-7P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and substitution reaction with dppe)
IT 406462-94-0P
RL: CAT (Catalyst use); PRP (Properties); SPN (Synthetic preparation); PREP (Preparation); USES (Uses)
(preparation of)
IT 406462-96-2P 406462-97-3P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
IT 406462-93-9P
RL: CAT (Catalyst use); PRP (Properties); SPN (Synthetic preparation); PREP (Preparation); USES (Uses)
(preparation, crystal structure, and attempted catalyst for oxygen atom transfer)
IT 74-85-1, Ethylene, reactions 7732-18-5, Water, reactions 172800-85-0
406462-90-6 406462-98-4
RL: RCT (Reactant); RACT (Reactant or reagent)
(water-promoted reaction of a platinum oxo complex with ethylene)
AB Treatment of [(dppp)Pt(μ -O)]₂(LiOTf)₂ (dppp = Ph₂P(CH₂)₃Ph₂) with ethylene in the presence of trace amts. of water results in oxygen atom transfer to one arm of the bidentate phosphine ligand and formation of [(dpppO)Pt(η -CH₂:CH₂)₂ (dpppO = Ph₂P(CH₂)₃P(O)Ph₂). Further investigation reveals that the reaction of [L₂P(μ -O)]₂(LiOTf)₂ (L = dppp, dppb) with water forms (L)₂Pt(OH)₂, which acts as a catalyst for the oxygen atom transfer reaction. The analogous oxo complex [(PPh₃)₂Pt(μ -O)]₂(LiBF₄)₂ does not react with ethylene under similar conditions. These results indicate that hydroxo complex intermediates should be considered in oxygen atom transfer reactions.
REFERENCE COUNT: 42 THERE ARE 42 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

ACCESSION NUMBER: 1998:625562 CAPLUS <<LOGINID:20080918>>
 DOCUMENT NUMBER: 129:339029
 ORIGINAL REFERENCE NO.: 129:68921a,68924a
 TITLE: Phosphorus oxonitridosodalites: synthesis using a molecular precursor and structural investigation by x-ray and neutron powder diffraction and ³¹P MAS NMR spectroscopy
 AUTHOR(S): Stock, Norbert; Irran, Elisabeth; Schnick, Wolfgang
 CORPORATE SOURCE: Laboratorium Anorganische Chemie Universität, Bayreuth, D-95440, Germany
 SOURCE: Chemistry--A European Journal (1998), 4(9), 1822-1828
 CODEN: CEUJED; ISSN: 0947-6539
 PUBLISHER: Wiley-VCH Verlag GmbH
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 TI Phosphorus oxonitridosodalites: synthesis using a molecular precursor and structural investigation by x-ray and neutron powder diffraction and ³¹P MAS NMR spectroscopy
 IT Crystal structure
 Molecular structure
 (of copper and lithium phosphorus oxonitridosodalites)
 IT 13597-72-3, Phosphoric triamide
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for preparation of copper and lithium phosphorus oxonitridosodalites)
 IT 7664-41-7, Ammonia, reactions 7783-20-2, Ammonium sulfate ((NH₄)₂SO₄), reactions 10026-13-8, Phosphorus chloride (PCl₅)
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for preparation of copper and lithium phosphorus oxonitridosodalites using a mol. precursor)
 IT 13966-08-0P 182505-10-8P
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
 (for preparation of copper and lithium phosphorus oxonitridosodalites using a mol. precursor)
 IT 7758-89-6, Copper chloride (CuCl) 22722-08-3, Phosphenimidic nitride
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for preparation of copper phosphorus oxonitridosodalite)
 IT 10377-51-2, Lithium iodide (LiI) 12124-97-9, Ammonium bromide 12136-58-2, Lithium sulfide (Li₂S) 13455-05-5, Phosphorothioic triamide
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for preparation of lithium phosphorus oxonitridosodalite)
 IT 7447-41-8, Lithium chloride, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for preparation of lithium phosphorus oxonitridosodalite using a mol. precursor)
 IT 215191-22-3P 215191-26-7P 215191-27-8P
 215191-28-9P
 RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
 (preparation and crystal structure)
 AB The oxonitridophosphates M⁸-mHm[P₁₂N₁₈O₆]Cl₂ (M = Cu, Li) with a sodalite-like [P₁₂N₁₈O₆]6- framework of corner-sharing PN₃O tetrahedra were synthesized by the reaction of the resp. metal chlorides with (NH₂)₂P(O)NP(NH₂)₃-NH₄Cl. In this precursor the desired molar ratio, P:O = 2:1, of the [P₁₂N₁₈O₆]6- framework structure was preorganized on a mol. level. Analogous oxonitridosodalites also were obtained from the metal salts MX (M = Cu, Li; X = Cl, Br, I) or Li₂S, the P/O and P/N components OP(NH₂)₂3 or HPN₂, and NH₄X or MX as halogen sources. The crystal structures of the phosphorus oxonitridosodalites Cu₄.8H₃.2[P₁₂N₁₈O₆]Cl₂ (1), Li₅.5H₂.5[P₁₂N₁₈O₆]Cl₂ (2), Li₆.2H₁.8[P₁₂N₁₈O₆]Br₂ (3), and Li₅.8H₂.2[P₁₂N₁₈O₆]I₂ (4) were studied by using neutron and x-ray powder diffraction as well as ³¹P MAS NMR

spectroscopy. Rietveld refinements were performed in the cubic space group I.hivn.43m ($Z = 1$, $a = 820.25(1)$ to $830.81(2)$ pm for $X = \text{Cl}$ to I). No exptl. evidence for a crystallog. ordering of the N/O atoms and for other than PN30 tetrahedra in the sodalite frameworks was obtained.

REFERENCE COUNT: 35 THERE ARE 35 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 8 OF 18 CAPLUS COPYRIGHT 2008 ACS ON STN

ACCESSION NUMBER: 1996:279252 CAPLUS <<LOGINID:20080918>>

DOCUMENT NUMBER: 125:24809

ORIGINAL REFERENCE NO.: 125:4671a,4674a

TITLE: Co-ordination chemistry of tridentate phosphinothiolates; syntheses and structures of $[\text{Li4}\{\text{PhP}(\text{C6H3S-2-SiMe3-3})2\}2(\text{MeOCH2CH2OMe})2]$, $[\text{Sn2}\{\text{PhP}(\text{C6H3S-2-SiMe3-3})2\}2]$ and $[\text{NHET3}][\text{Sm}\{\text{PhP}(\text{C6H3S-2-R-3})2\}2(\text{py})2]\cdot 2\text{py}$ ($\text{R} = \text{H}$ or SiMe3 , $\text{py} = \text{C5H5N}$)

AUTHOR(S): Froelish, Nikolaus; Hitchcock, Peter B.; Hu, Jin;

CORPORATE SOURCE: Lappert, Michael F.; Dilworth, Jonathan R. Sch. Chem. Molecular Sciences, Univ. Sussex, Brighton, BN1 9QJ, UK

SOURCE: Journal of the Chemical Society, Dalton Transactions: Inorganic Chemistry (1996), (9), 1941-1946 CODEN: JCDTBI; ISSN: 0300-9246

PUBLISHER: Royal Society of Chemistry

DOCUMENT TYPE: Journal

LANGUAGE: English

TI Co-ordination chemistry of tridentate phosphinothiolates; syntheses and structures of $[\text{Li4}\{\text{PhP}(\text{C6H3S-2-SiMe3-3})2\}2(\text{MeOCH2CH2OMe})2]$, $[\text{Sn2}\{\text{PhP}(\text{C6H3S-2-SiMe3-3})2\}2]$ and $[\text{NHET3}][\text{Sm}\{\text{PhP}(\text{C6H3S-2-R-3})2\}2(\text{py})2]\cdot 2\text{py}$ ($\text{R} = \text{H}$ or SiMe3 , $\text{py} = \text{C5H5N}$)

IT Crystal structure

Molecular structure

(of lithium and tin and samarium phenylbis(mercaptophenyl)phosphine complexes)

IT 119327-23-0

RL: RCT (Reactant); RACT (Reactant or reagent) (for preparation of lithium and tin and samarium phenylbis(mercaptophenyl)phosphine complexes)

IT 177594-36-4, Triiodobis(tetrahydrofuran)samarium

RL: RCT (Reactant); RACT (Reactant or reagent) (for preparation of samarium phenylbis(mercaptophenyl)phosphine complexes)

IT 119327-18-3

RL: RCT (Reactant); RACT (Reactant or reagent) (for preparation of samarium phenylbis(mercaptophenyl)phosphine pyridine complex)

IT 177594-29-5DP, reaction products with ytterbium iodide

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (preparation and NMR of)

IT 177594-29-5P 177594-31-9P 177594-33-1P 177594-35-3P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (preparation and crystal structure of)

IT 19357-86-9DP, Ytterbium diiodide, reaction product with lithium phenylbis(mercaptophenyl)phosphine complex

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (preparation of)

AB Treatment of $\text{PhP}(\text{C6H3}(\text{SH-2})\text{R-3})2$ ($\text{R} = \text{H}$ (H2L1) or SiMe3 (H2L2)) with SmI3 and NET3 and then recrystn. from pyridine (py) yielded $[\text{NHET3}][\text{SmL2}(\text{py})2]\cdot 2\text{py}$ ($\text{L} = \text{L1}$ (3) or L2 (4)), while H2L2 and LiBu or $[\text{Sn}(\text{N}(\text{SiMe3})2)2]$ afforded $[\{\text{Li2L2}(\text{dme})\}2]$ (1) (after addition of dme) or $[\{\text{SnL2}\}2]$ (2), resp. ($\text{dme} = \text{MeOCH2CH2OMe}$). 1-4 Were characterized by

microanal., NMR spectra and single-crystal x-ray diffraction.

L6 ANSWER 9 OF 18 CAPLUS COPYRIGHT 2008 ACS ON STN
ACCESSION NUMBER: 1994:218017 CAPLUS <<LOGINID:20080918>>
DOCUMENT NUMBER: 120:218017
ORIGINAL REFERENCE NO.: 120:38729a,38732a
TITLE: Chiral tripod ligand: one pot reaction MeC(CH₂PPh₂)₃
→ MeC(CH₂P(Ph)(R))₃; intermediate,
diastereoselective control and complex chemistry
AUTHOR(S): Walter, Olaf; Klein, Thomas; Huttner, Gottfried;
Zsolnai, Laszlo
CORPORATE SOURCE: Anorganisch-Chemisches Institut der Universitaet
Heidelberg, Im Neuenheimer Feld 270, Heidelberg, 6900,
Germany
SOURCE: Journal of Organometallic Chemistry (1993), 458(1-2),
63-81
CODEN: JORCAI; ISSN: 0022-328X
DOCUMENT TYPE: Journal
LANGUAGE: German
OTHER SOURCE(S): CASREACT 120:218017
TI Chiral tripod ligand: one pot reaction MeC(CH₂PPh₂)₃ →
MeC(CH₂P(Ph)(R))₃; intermediate, diastereoselective control and complex
chemistry
IT Crystal structure
Molecular structure
(of chiral tripod phosphorus ligand molybdenum complexes)
IT 22031-12-5
RL: RCT (Reactant); RACT (Reactant or reagent)
(lithiation and sequential alkylation or molybdenum complexation of)
IT 79190-90-2P 153956-06-OP 153956-43-5P 154006-92-5P
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(preparation and crystal structure of)
IT 153956-39-9P 153956-40-2P 154006-91-4P 154096-26-1P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and multinuclear NMR of)
IT 153956-41-3P 153956-42-4P 154006-93-6P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
IT 145011-41-2P 145033-21-2P 153888-54-1P 153888-55-2P 153888-56-3P
153888-57-4P 154002-80-9P 154002-81-0P 154002-82-1P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation, NMR, and complexation of, with molybdenum)
IT 153956-44-6P 154005-24-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation, mol. structure, or alkylation of)
IT 15038-48-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with chiral tripod phosphorus ligand)
AB The tripod ligand H₃CC(CH₂PPh₂)₃, 1, reacts with lithium metal to produce
H₃CC(CH₂PPhLi)₃, 2, in good yields. Compound 2 crystallizes in the form of
2·4THF·MeN(CH₂CH₂NMe)₂. The crystal structure of this
adduct shows bridging as well as terminal phosphorus-coordination of
lithium. Compound 2 reacts with electrophiles RX to give
H₃CC(CH₂P(Ph)(R))₃, 3 (R = H, Me, Et, CHMe₂, CH₂Ph, 3a-3e). The
transformation 1 → 2 → 3 can be performed in a one-pot
reaction. While the chiral compds. 3 may be characterized as such, their
reaction with (CH₃CN)Mo(CO)₃ yields the easy-to-characterize
coordination compds. H₃CC(CH₂P(Ph)(R))₃Mo(CO)₃, 5. NMR anal. of
3 and 5 shows that from the two diastereomeric forms of 3 (the
enantiomeric pair SSR/RRS and the pair SSS/RRR) only the
SSR/RRS-diastereomer is formed with R = CH₂Ph (3e, 5e). In the other

cases (R = H, Me, Et, CHMe2) a statistical 3 : 1 mixture of the two diastereomers is formed. These findings are further corroborated by x-ray analyses of H3CC(CH2P(Ph)(Et))3Mo(CO)3, 5c, and H3CC(CH2P(Ph)(CH2Ph))3Mo(CO)3, 5e. The remarkable facts reported in this paper are the ease with which the chiral tripod ligands H3CC(CH2P(Ph)(R))3, 3, are prepared, and the evidence that their formation may well involve diastereoselective control.

L6 ANSWER 10 OF 18 CAPLUS COPYRIGHT 2008 ACS ON STN
 ACCESSION NUMBER: 1993:449648 CAPLUS <<LOGINID:20080918>>
 DOCUMENT NUMBER: 119:49648
 ORIGINAL REFERENCE NO.: 119:9021a,9024a
 TITLE: Contributions to the chemistry of boron. 214.
 Synthesis and reaction chemistry of
 aminophosphinoboranes
 AUTHOR(S): Dou, Danan; Westerhausen, Matthias; Wood, Gary L.;
 Linti, Gerald; Duesler, Eileen N.; Noth, Heinrich;
 Paine, Robert T.
 CORPORATE SOURCE: Dep. Chem., Univ. New Mexico, Albuquerque, NM, 87131,
 USA
 SOURCE: Chemische Berichte (1993), 126(2), 379-97
 CODEN: CHBEAM; ISSN: 0009-2940
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 119:49648
 TI Contributions to the chemistry of boron. 214. Synthesis and reaction
 chemistry of aminophosphinoboranes
 IT Ring closure and formation
 (of aminophosphinoboranes)
 IT Crystal structure
 Molecular structure
 (of aminophosphinoboranes, diphosphadiboretanes and
 triphosphatriborinanes)
 IT 148446-90-6P 148446-91-7P 148446-92-8P
 RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
 (preparation and crystal and mol. structure of)
 IT 112438-36-5P
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
 (Reactant or reagent)
 (preparation and reaction of, with lithium disilylphosphide)
 IT 112438-35-4P
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
 (Reactant or reagent)
 (preparation and reactions of)
 IT 147875-93-2P
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
 (Reactant or reagent)
 (preparation and thermal intramol. cyclization of)
 IT 112438-37-6P 122501-56-8P 147875-75-0P 147875-76-1P 147875-77-2P
 147875-78-3P 147875-79-4P 147875-80-7P 147875-81-8P 147875-82-9P
 147875-83-0P 147875-84-1P 147875-86-3P 147875-87-4P 147875-89-6P
 147875-90-9P 147875-91-0P 147875-92-1P 147875-95-4P
 148446-93-9P 148446-94-0P 148446-95-1P
 RL: SPN (Synthetic preparation); PREP (Preparation)
 (preparation of)
 IT 147875-88-5P
 RL: SPN (Synthetic preparation); PREP (Preparation)
 (preparation, crystal and mol. structure, and intramol. cyclization of)
 IT 147875-94-3P
 RL: SPN (Synthetic preparation); PREP (Preparation)
 (preparation, crystal and mol. structure, and reaction of, with methanol)

IT 122528-73-8P 147875-85-2P
 RL: SPN (Synthetic preparation); PREP (Preparation)
 (preparation, crystal and mol. structure, and reaction of, with
 pentacarbonylchromium-trimethylamine complex)

IT 59610-41-2 62263-24-5
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with aminodichloroboranes)

IT 15228-26-9 15228-32-7
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with aminophosphinoboranes)

IT 868-30-4 1113-31-1 1139-65-7 6591-26-0 44873-49-6 79855-29-1
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with lithium phosphides)

AB Aminochlorophosphinoboranes (R2N)B(Cl)P(SiMe3)2 and (R2N)B(Cl)PH2 are
 obtained from elimination reactions between aminochloroboranes and
 LiP(SiMe3)2 and LiPH2, resp. Selected reaction chemical of
 (i-Pr2N)B(Cl)P(SiMe3)2 with NH3, Me3SiN3, Cr(CO)
)5-NMe3, and W(CO)5-NMe3 is described.
 The azide (i-Pr2N)B(N3)P(SiMe3)2 is stable at 25°; however,
 thermolysis at 80° provides a novel six-membered ring compound
 [(i-Pr2N)BN(SiMe3)P(SiMe3)]2. The reaction of (Ph2N)B(Cl)P(SiMe3)2 with
 LiP(SiMe3)2 produces the only isolable bis(phosphino)borane, while
 combination of (R2N)B(Cl)PH2 with LiPH2·DME yields new
 diphosphadiboretanes [(R2N)BPH]2 (R2N = i-Pr2N, Ph2N, tmp =
 2,2,6,6-tetramethylpiperidino) and triphosphatriborinanes [(R2N)BPH]3 [R2N
 = (Me3Si)2N, Me2N, Et2N]. Two salts, [(i-Pr2N)BP(H)B(i-
 Pr2N)PLi·DME]2 and [tmpBP(H)B(tmp)PLi·DME]2 (DME = ethylene
 glycol di-Me ether) are also isolated. The results of mol. structure
 detns. for [(i-Pr2N)BN(SiMe3)P(SiMe3)]2, [tmpBPH]2, [tmpBPH]2·
 Cr(CO)5, [(Me3Si)2N]BPH]3, [(Me3Si)2N]BPH]3·
 Cr(CO)5, (Ph2N)B[P(SiMe3)2]2, and [(i-Pr2N)BP(H)B(i-
 Pr2N)PLi·DME]2 by x-ray crystallog. are discussed.

L6 ANSWER 11 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN
 ACCESSION NUMBER: 1992:511748 CAPLUS <<LOGINID:20080918>>
 DOCUMENT NUMBER: 117:111748
 ORIGINAL REFERENCE NO.: 117:19511a,19514a
 TITLE: Tetraphosphafulvalene dianions isoelectronic to
 tetrathiafulvalene dications

AUTHOR(S): Maigrot, Nicole; Ricard, Louis; Charrier, Claude;
 Mathey, Francois

CORPORATE SOURCE: Lab. Chim. Phosphore Met. Transition, Ec. Polytech.,
 Palaiseau, F-91128, Fr.

SOURCE: Angewandte Chemie (1992), 104(8), 1082-4 (See also
 Angew. Chem., Int. Ed. Engl., 1992, 31(8), 1031-2)
 CODEN: ANCEAD; ISSN: 0044-8249

DOCUMENT TYPE: Journal
 LANGUAGE: German
 OTHER SOURCE(S): CASREACT 117:111748

TI Tetraphosphafulvalene dianions isoelectronic to tetrathiafulvalene
 dications

IT Crystal structure
 Molecular structure
 (of tetraphosphafulvalene dianion and its reaction product with sulfur)

IT 114862-89-4
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (metalation of, with lithium or potassium, dianions by)

IT 142397-66-8P 142397-69-1P
 RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
 (preparation and crystal structure of)

IT 142397-61-3P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
 (preparation and mol. structure of)

IT 142397-60-2P
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
 (Reactant or reagent)
 (preparation and reaction of, with sulfur or iron complex)

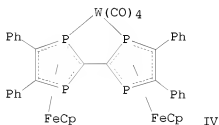
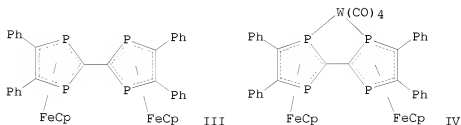
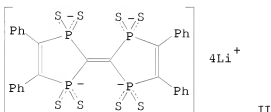
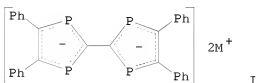
IT 142397-67-9P
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
 (Reactant or reagent)
 (preparation and reaction of, with tungsten complex)

IT 142397-59-9P 142397-68-0P
 RL: SPN (Synthetic preparation); PREP (Preparation)
 (preparation of)

IT 34978-37-5
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with tetraphosphafulvalene dianion)

IT 12129-25-8
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with tetraphosphafulvalene dianion complex)

GI



AB Metalation of an octaphenyl derivative of tetraphosphafulvalene with Li or K metal in THF gave tetraphosphafulvalene dianions I (M = Li, K). Reaction of I (M = Li) with excess of sulfur gave lithium tetrakis(dithiophosphinate) derivative II. Reaction of I (M = Li) with (η5-cyclopentadienyl)(η6-xylene)iron hexafluorophosphate, [(Cp)(η6-1,4-Me2C6H4) Fe] + PF6-, gave 30% iron complex III which on further reaction with [(nbd)W(CO)4] (nbd = norbornadiene) gave tungsten complex IV. The crystal structures of I (M =

K) and II were determined I are isoelectronic to tetrathiafulvalene dications.

L6 ANSWER 12 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1991:66017 CAPLUS <<LOGINID::20080918>>
DOCUMENT NUMBER: 114:66017
ORIGINAL REFERENCE NO.: 114:11249a,11252a
TITLE: Structural hierarchy in M[6]T[4].vphi.n minerals
AUTHOR(S): Hawthorne, Frank C.
CORPORATE SOURCE: Dep. Geol. Sci., Univ. Manitoba, Winnipeg, MB, R3T 2N2, Can.
SOURCE: Zeitschrift fuer Kristallographie (1990), 192(1-2), 1-52
CODEN: ZEKRDZ; ISSN: 0044-2968
DOCUMENT TYPE: Journal
LANGUAGE: English
TI Structural hierarchy in M[6]T[4].vphi.n minerals
IT Minerals
RL: PRP (Properties)
(classification of, into crystal-structure hierarchy group based on bond valence theory)
IT 1302-58-5, Amblygonite 1318-31-6, Clinozoisite 1318-45-2, Dickite 1318-49-6, Epidote 1318-72-5, Kainite 1318-74-7, Kaolinite, properties 1318-81-6, Lawsonite 1319-12-6, Pumpellyite 1319-39-7, Roesslerite 1319-42-2, Zoisite 11089-72-8, Varulite-NaNa (Mn1-1.5Fe0.5-1)MnNa(Na0.5-1Ca0-0.5)(PO4)3 12004-28-3, Palermoite 12006-42-7, Arthurite 12025-97-7, Uklonskovite 12053-86-0, Linarite 12062-75-8, Tavorite 12068-50-7, Halloysite 12135-61-4, Titanite 12172-70-2, Allanite 12172-72-4, Alluaudite 12174-22-0, Piemontite 12198-53-7, Lazulite 12198-84-4, Natromontebrazite 12206-95-0, Fibroferite 12211-29-9, Ardenite 12211-42-6, Scorzalite 12218-55-2, Leucophosphate 12244-16-5, Endellite 12260-00-3, Childrenite 12260-04-7, Montebrazite 12267-58-2, Paravauxite 12267-88-8, Isokite 12274-61-2, Hagedorffite-NaNa ((Fe0.5-1Mg0-0.5)1-1.5Fe0.5-1)MnNa(Na0.5-1Ca0-0.5)(PO4)3 12279-65-1, Nacrite 12284-84-3, Liroconite 12297-96-0, Strunzite 12313-62-1, Gabrielsonite 12325-36-9, Mottramite 12337-04-1, Amarantite 12343-52-1, Metavauxite 12350-25-3, Mitridatite 12413-59-1, Apowite 12414-10-7, Bermanite 12415-36-0, Eosphorite 12416-31-8, Hancockite 12416-52-3, Holdenite 12417-43-5, Lacroixite 12417-80-0, Morinite 12417-91-3, Veszelyite 12418-01-8, Durangite 12418-22-3, Gordonite (Mg[Al(OH)(PO4)]2.8H2O) 12418-52-9, Arseniosiderite 12418-83-6, Chlorothionite 12418-91-6, Butlerite 12418-92-7, Hohmannite 12419-94-2, Minyulite 12420-03-0, Moorhouseite 12420-18-7, Nickel hexahydrite 12421-00-0, Pseudolaueite 12424-68-9, Sigloite 12426-43-6, Tilasite 12426-77-6, Parabutlerite 12518-72-8, Stewartite 12601-23-9, Julgoldite 13587-33-2, Poitevinite 13759-09-6, Morenosite 13778-97-7, Hexahydrite 13817-21-5, Chalcantite 13824-49-2, Strengite 13824-50-5, Variscite 14457-55-7, Epsomite 14567-64-7, Kieserite 14567-68-1, Szomolnokite 14567-70-5, Sideritol 14567-75-0, Phosphosiderite 14567-77-2, Plancheite 14654-06-9, Clinoenstatite 14654-10-5, Phosphorosslerite (Mg(HPO4).7H2O) 14654-11-6, Newberyite 14654-14-9, Kolbeckite 14681-78-8, Orthoenstatite 14752-50-2, Metavariscite 15230-85-0, Clinoferrrosilite 15318-72-6, Laueite 15422-36-3, Orthoferrrosilite 15490-91-2, Struvite 15491-15-3, Goslarite 15491-23-3, Melantherite 15553-21-6, Pentahydrite 15606-25-4, Dioptase 15652-21-8, Barbasolite 15669-02-0, Malayaite 16094-11-4, Conicalcite 16263-38-0, Austinite 17068-62-1, Hypersthene 19004-61-6, Descloizite 20775-42-2, Retgersite 20909-44-8, Scordite 21793-55-5, Mansfieldite 22206-42-4, Calciovolborthite 23540-24-1, Pyrobelonite 23625-76-5, Adelite 24189-48-8, Gunningite 24189-58-0, Clinohypersthene 24189-61-5, Duftite 24250-37-1, Bieberite (Co(SO4).7H2O) 24250-39-3,

Bonattite 24250-46-2, Carminite 24250-50-8, Bianchite 24378-28-7,
 Boothite 24378-31-2, Starkeyite 33636-44-1, Tsumcorite 39473-77-3,
 Melonjosephite 51184-69-1, Jagowerite (Ba[Al(OH)(PO4)]2) 51198-90-4,
 Robertsite 51340-44-4, Khademite 52717-37-0, Szmikite 53125-64-7,
 Brassite 54652-49-2, Whitmoreite 58206-65-8, Foggite 59042-33-0,
 Krautite (Mn(HAsO4).H2O) 59492-33-0, Jurbanite 60867-12-1,
 Vuagnatite 61026-43-5, Bertossaite 64476-46-6, Rozenite 66183-78-6,
 Ferroxahydrite 66457-84-9, Ferropumpellyite 67712-23-6, Boyleite
 68108-22-5, Jokokuite (Mn(SO4).5H2O) 70315-78-5, Ilesite
 70878-67-0, Koritnigite 72276-48-3, Maghagendorffite-Na
 ((Mg0.5-1Fe0-0.5)1-1.5Fe0.5-1)[Mn(Na0.5-1Ca0-0.5)](PO4)2(HPO4)]
 73347-41-8, Mallardite 73729-55-2, Zinc-melanterite 74410-63-2,
 Fluckite 75137-72-3, Schmiederite 75797-73-8, Medaite 75797-77-2,
 Tiragalloite 79078-56-1, Cobalt-koritnigite 80147-36-0, Ojuelaite
 80487-65-6, Cechite 80487-70-3, Panasqueiraite 80487-72-5, Shuiskite
 83061-39-6, Arsendesclizite 83061-40-9, Kolfanite 83380-55-6,
 Dwornikite 85712-31-8, Ushkovite 88201-08-5, Ferrostrunzite
 93442-96-7, Earlshannonite 127030-85-7, Allanite-(Y)
 RL: PRP (Properties)

(classification of, into crystal-structure hierarchy group based on
 bond valence theory)

AB A large number of minerals have the general stoichiometry
 $A_x[M_6]T[4].vphi.n.y.vphi.z$, where A are large high-coordination number
 cations such as alkali metals and alkaline earths, M[6] are [6]-coordinate
 divalent to quadrivalent cations, T[4] are [4]-coordinate trivalent to
 hexavalent cations, and .vphi. are unspecified simple anions. The square
 brackets denote the more strongly bonded part of the structure, called the
 structural unit. This way of expressing a structural formula essentially
 gives a binary representation of the structure, whereby the structural
 unit can be considered as a very complex oxyanion that interacts with the
 A species that constitute the cationic part of the structure; the .vphi.
 anions outside the square brackets are anions only very weakly held in the
 structure. There are 2 important features of expressing the formula in
 such a manner: very complex interactions within the structure are reduced
 to a simple binary interaction that is susceptible to quant. anal. using
 bond-valence theory; an hierarchical structural scheme may be set up by
 considering the graphical/topol. properties of the structural unit. Such
 a structure hierarchy is set up for minerals with structural units of the
 general formula $[M_6]T[4].vphi.n$, based on the hypothesis that crystal
 structures may be ordered according to the polymerization of the coordination
 polyhedra of higher bond-valences. The structures are arranged into
 groups according to the dimension of polymerization of the structural unit and
 are arranged within these groups in terms of increasing d.p. There is
 definitely a preferred sequence of polyhedral linkage with increasing
 degree of condensation; from a completely disconnected structural unit,
 there is linkage between octahedra and tetrahedra, followed by linkage
 between octahedra, followed by linkage between tetrahedra. Along with
 this is a systematic change in the stoichiometry of the structural unit;
 this suggests that in a chemical formula, there is much more structural
 information than currently realized. The possible clusters of
 $[M_6]2T[4]2.vphi.n$ stoichiometry are derived using graph theoretic and
 combinatorial techniques, subject to the boundary conditions that certain
 polyhedral linkages (e.g. face-sharing between tetrahedra) will not occur.
 There are 76 completely connected clusters of the form $[M_6]2T.vphi.n$, but
 only 6 are found as fundamental building blocks in the structures of the
 96 minerals considered.

TITLE: Alkali metal phosphorus compounds and their reactions.
XXI. Reaction of potassium ethyl phosphide with dihaloalkanes

AUTHOR(S): Issleib, K.; Doell, G.

CORPORATE SOURCE: Martin-Luther Univ., HalleSaale, Germany

SOURCE: Zeitschrift fuer Anorganische und Allgemeine Chemie (1963), 324(5-6), 259-69
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DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

TI Alkali metal phosphorus compounds and their reactions. XXI. Reaction of potassium ethyl phosphide with dihaloalkanes

IT Insecticides
(dialkyl [2,2-dichloro-1-(dichloromethyl)-1-hydroxyethyl]phosphonates as)

IT Baptisia tinctoria
(glycosides of)

IT Paraffins
(α,ω -dihalo, reaction with (ethylphosphino)-potassium)

IT Ethyl ether, compound with [tetramethylenebis(ethylphosphinothioylidene)]dilithium
Phosphinodithioic acid, hexamethylenebis[ethyl-, cobalt salt
RL: PREP (Preparation)

IT 2404-55-9 93905-92-1
(Derived from data in the 7th Collective Formula Index (1962-1966))

IT 593-68-0, Phosphine, ethyl-
(potassium derivative, reaction with α,ω -dihaloalkanes)

IT 3040-70-8P, Tetraphosphetane, tetraethyl- 7789-43-7P, Cobalt bromide, CoBr₂, compds. with tetramethylenebis[ethylphosphine] 7789-43-7P, Cobalt bromide, CoBr₂, compds. with pentamethylenebis[ethylphosphine] 20626-76-0P, Tetraphosphetanium, 1,2,3,4-tetraethyl-1-methyl-, iodide 34616-20-1P, Phosphonic acid, [2,2-dichloro-1-(dichloromethyl)-1-hydroxyethyl]-, dimethyl ester 89798-26-5P, Phosphine, methylenebis[ethyl- 90274-10-5P, Phosphinodithioic acid, tetramethylenebis[ethyl- 90948-90-6P, Phosphonic acid, [2,2-dichloro-1-(dichloromethyl)-1-hydroxyethyl]-, diisopropyl ester 91065-37-1P, Phosphinodithioic acid, hexamethylenebis[ethyl- 91369-40-3P, Phosphonic acid, [2,2-dichloro-1-(dichloromethyl)-1-hydroxyethyl]-, dibutyl ester 91369-41-4P, Phosphonic acid, [2,2-dichloro-1-(dichloromethyl)-1-hydroxyethyl]-, diisobutyl ester 91469-51-1P, Phosphine sulfide, methylenebis[ethyl- 91772-46-2P, Phosphine, trimethylenebis[ethyl- 91772-47-3P, Phosphine sulfide, trimethylenebis[ethyl- 92045-94-8P, Phosphine, tetramethylenebis[ethyl- 92156-35-9P, Phosphonic acid, [2,2-dichloro-1-(dichloromethyl)-1-hydroxyethyl]-, diisopentyl ester 92381-19-6P, Phosphine, trimethylenebis[ethyl-, compound with HI 92381-19-6P, Hydriodic acid, compound with trimethylenebis[ethylphosphine] (2:1) 92400-37-8P, Phosphine, tetramethylenebis[ethyl-, compound with HI 92400-37-8P, Hydriodic acid, compound with tetramethylenebis[ethylphosphine] (2:1) 92443-15-7P, Phosphine sulfide, tetramethylenebis[ethyl- 92443-27-1P, Phosphine oxide, tetramethylenebis[ethyl- 93284-04-9P, Phosphine sulfide, pentamethylenebis[ethyl- 93284-52-7P, Phosphinodithioic acid, pentamethylenebis[ethyl-, nickel salt 93335-48-9P, Phosphine, pentamethylenebis[ethyl-, compound with HI 93456-93-0P, Phosphine, trimethylenebis[ethylmethyl-, compound with HI 93481-01-7P, Phosphine, pentamethylenebis[ethyl- 94073-52-6P, Hydriodic acid, compound with hexamethylenebis[ethylphosphine] 94073-52-6P, Phosphine, hexamethylenebis[ethyl-, compound with HI 94073-53-7P, Phosphine, tetramethylenebis[ethylmethyl-, compound with HI 94074-49-4P, Phosphine, hexamethylenebis[ethyl- 94115-74-9P, Phosphine, tetramethylenebis[ethyl-, compound with CoBr₂ 94315-15-8P, Phosphine, pentamethylenebis[ethyl-,

compound with CoBr₂ 95322-84-2P, Phosphine, hexamethylenebis[ethylmethyl-, compound with HI 96117-38-3P, Phosphine, pentamethylenebis[ethylmethyl-, compound with HI 97340-94-8P, Lithium, [trimethylenebis(ethylphosphinidene)]di-, compound with p-dioxane 98220-41-8P, Lithium, [tetramethylenebis(ethylphosphinidene)]di-, compound with p-dioxane 98338-80-8P, Lithium, [pentamethylenebis(ethylphosphinidene)]di-, compound with p-dioxane 105667-64-9P, Lithium, [hexamethylenebis(ethylphosphinidene)]di-, compound with p-dioxane 108652-58-0P, Lithium, [tetramethylenebis(ethylphosphinothioylidene)]di-, compound with Et₂O 820992-86-7P, Hydriodic acid, compound with pentamethylenebis[ethylphosphine] 859043-27-9P, Isopentyl alcohol, [2,2-dichloro-1-(dichloromethyl)-1-hydroxyethyl]phosphonate 859455-66-6P, Hydriodic acid, compound with tetramethylenebis[ethylmethylphosphine] (2:1) 860390-77-8P, Hydriodic acid, compound with trimethylenebis[ethylmethylphosphine] 860390-79-0P, Hydriodic acid, compound with pentamethylenebis[ethylmethylphosphine] 860390-81-4P, Hydriodic acid, compound with hexamethylenebis[ethylmethylphosphine]

RL: PREP (Preparation)
(preparation of)

IT 123-91-1P, p-Dioxane
RL: PREP (Preparation)
(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [hexamethylenebis(ethylphosphinidene)]dilithium (2:1))

IT 123-91-1P, p-Dioxane
RL: PREP (Preparation)
(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [pentamethylenebis(ethylphosphinidene)]dilithium (2:1))

IT 123-91-1P, p-Dioxane
RL: PREP (Preparation)
(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [tetramethylenebis(ethylphosphinidene)]dilithium (2:1))

IT 123-91-1P, p-Dioxane
RL: PREP (Preparation)
(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [trimethylenebis(ethylphosphinidene)]dilithium (2:1))

IT 88983-00-0, Potassium, (ethylphosphino)-
(reaction with α,ω -dihaloalkanes)

IT 7440-48-4, Cobalt
(salts, of hexamethylenebis[ethylphosphinodithioic acid])

IT 7440-02-0, Nickel
(salts, of pentamethylenebis[ethylphosphinodithioic acid])

IT 92443-15-7, Phosphine sulfide, tetramethylenebis[ethyl-
(P,P'-dilithium derivative, compound with Et₂O)

IT 91772-46-2, Phosphine, trimethylenebis[ethyl- 92045-94-8, Phosphine, tetramethylenebis[ethyl- 93481-01-7, Phosphine, pentamethylenebis[ethyl- 94074-49-4, Phosphine, hexamethylenebis[ethyl-
(P,P'-dilithium derivative, compound with p-dioxane)

AB cf. CA 59, 5191f. Di-secondary phosphines, EtHP(CH₂)_nPHet (n = 3-6), chelate complex ligands, are formed by treatment of KPHEt with alkylenedihalides. These phosphines react with PhLi, MeI, and HI to form Et(Li)P[CH₂]nP(Li)Et, [MeEtHP(CH₂)_nPHetMe]++ 2I-, and [EtH₂P(CH₂)_nPH₂Et]++ 2I-, resp. KPHEt (20.5 g.) was prepared from 20 g. EtPH₂ and 8.5 g. K in 500 ml. Pr₂O at 80-85°. A suspension of 20 g. KPHEt in Pr₂O was treated with 22 g. 1,6-dibromohexane in 500 ml. Pr₂O to yield 12 g. EtHP(CH₂)₆PHet, b₂ 92°. Similarly prepared were EtHP(CH₂)₅PHet (12.3 g.), b₂ 79° EtHP(CH₂)₄PHet (12.7 g.), b₁₆ 119°, and EtHP(CH₂)₃PHet (9.1 g.), b₁₇ 103°. Each of these di-secondary phosphines (2 g.) was dissolved in 20 ml. ether and treated with the corresponding amts. of MeI (3-4 g.) to give the resp. phosphonium salts, m. 110-12° 98-100°, 115-17° and 108-10°. The

same di-secondary phosphines (2 g.), each dissolved in 20 ml. ether, treated with 2 ml. concentrated aqueous HI gave corresponding phosphonium compds., m. 99-110°, 86-8° 102-3° and 116-18°, resp. Solns. of the di-secondary phosphines (2 g.) in 5 ml. dioxane (C4H8O2) were treated by dropwise addition of calculated amts. (16, 17, 19 and 20 ml., resp.) of PhLi solution (1 ml. = 94 mg.). The yields of colorless, difficultly soluble, extremely air- and moisture-sensitive products were: Et(Li)P(CH2)6P(Li)Et.2C4H8O2, 3.6 g.; Et(Li)P(CH2)5P(Li)Et.2C4H8O2, 3.3 g.; Et(Li)P(CH2)4P(Li)Et.2C4H8O2, 3.5 g.; and Et(Li)P(CH2)3P(Li)Et.2C4H8O2, 3.6 g. The following dithiophosphinic acids and phosphine sulfides were prepared by treating the corresponding di-secondary phosphines with various amts. of S in C6H6 solution: Et(HS)(S)P(CH2)6P(S)(SH)Et, m. 75-7°; EtH(S)P(CH2)6P(S)HET; EtH(S)P(CH2)4P(S)HET, m. 91-2°; Et(HS)(S)P(CH2)4P(S)(SH)Et, m. 125-7°; EtH(S)P(CH2)3P(S)HET, m. 86-7°. KPHEt (20 g.) in 500 ml. Pr2O treated with 8 g. CH2Cl2 in 50 ml. Pr2O gave only 0.7 g. EtHPCH2PhEt6, b5 54°. Other derivs. prepared for characterizing the various products were: [Et(S)(S)P(CH2)6P(S)(S)Et]--Ni++, decomposed 200° Et(Li)(S)P(CH2)4P(S)(Li)Et.4C4H10O; [Et(S)(S)P(CH2)6P(S)(S)Et]--Co++; EtH(O)P(CH2)4P(O)HET, m. 100-2°; EtHP(CH2)4PHET.CoBr2, decomposed 112-15°; EtHP(CH2)5PHET.CoBr2, decomposed 118-20°; (PET)4, b2 164-6°; [(PET)4Me]I, m. 84-6°; EtH(S)PCH2P(S)HET, m. 150-3°.

L6 ANSWER 14 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1963:428636 CAPLUS <<LOGINID:20080918>>

DOCUMENT NUMBER: 59:28636

ORIGINAL REFERENCE NO.: 59:5191f-h, 5192a-d

TITLE: Alkali metal phosphorus compounds and their reactions.

XX. 1,2-Ethylenebis(monocyclohexylphosphine) and -monoethylphosphine

AUTHOR(S): Issleib, Kurt; Doell, Gerhard

CORPORATE SOURCE: Martin-Luther-Univ., Halle, Germany

SOURCE: Chemische Berichte (1963), 96, 1544-50

CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE: Journal

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OTHER SOURCE(S): CASREACT 59:28636

TI Alkali metal phosphorus compounds and their reactions. XX.

1,2-Ethylenebis(monocyclohexylphosphine) and -monoethylphosphine

IT Alkali metal compounds

(phosphorus-containing organic)

IT Ethyl ether, compound of, with [ethylenebis(cyclohexylthiophosphinyldiene)dilithium (4:1)]

Phosphine sulfide, ethylenebis[cyclohexyl-, ±

RL: PREP (Preparation)

IT 18899-63-3, Phosphine, ethylenebis[cyclohexyl-

(P,P'-dilithium derivative)

IT 97001-21-3, Phosphine sulfide, ethylenebis[cyclohexyl-

(P,P'-dilithium derivative, compound with Et2O)

IT 18899-62-2, Phosphine, ethylenebis[ethyl-

(P,P'-dilithium derivative, compound with p-dioxane)

IT 7723-14-0, Phosphorus

(compds., alkali metal derivs. of organic)

IT 1571-33-1P, Phosphonic acid, phenyl- 2359-99-1P, Diphosphine,

tetracyclohexyl- 3040-63-9P, Diphosphine, tetraethyl- 6411-21-8P,

Phosphine, ethylenebis[diethyl- 6710-67-4P, Diphosphonium, pentaethyl-,

iodide 7237-16-3P, Phosphonic acid, phenyl-, diisopropyl ester

18899-61-1P, Phosphine, ethylenebis[cyclohexylmethyl-, compound with HI

23743-26-2P, Phosphine, ethylenebis[dicyclohexyl- 29149-95-9P,

p-Diphosporinane, 1,4-diethyl- 36540-03-1P, p-Diphosporinane, 1,4-dicyclohexyl- 91773-78-3P, Phosphine sulfide, ethylenebis[ethyl-92400-38-9P, Phosphine, ethylenebis[ethylmethyl-, compound with HI 94073-51-5P, Phosphine, ethylenebis[diethyl-, compound with HI 94626-87-6P, p-Diphosporinane, 1,4-diethyl-, 1,4-disulfide 96419-23-7P, Lithium, [ethylenebis(cyclohexylphosphinidene)]di- 96471-96-4P, Lithium, [ethylenebis(ethylphosphinidene)]di-, compound with p-dioxane 97001-21-3P, Phosphine sulfide, ethylenebis[cyclohexyl-, meso- 98000-09-0P, p-Diphosporinane, 1,4-dicyclohexyl-, 1,4-disulfide, cis- 98000-09-0P, p-Diphosporinane, 1,4-dicyclohexyl-, 1,4-disulfide, trans- 98282-75-8P, p-Diphosporinane, 1,4-dicyclohexyl-, 1,4-dioxide 100916-48-1P, Lithium, [ethylenebis(cyclohexylthiophosphinylidene)]di-, compound with Et2O 860390-83-6P, Hydriodic acid, compound with ethylenebis[ethylmethylphosphine] 860390-85-8P, Hydriodic acid, compound with ethylenebis[diethylphosphine] 860390-87-0P, Hydriodic acid, compound with ethylenebis[cyclohexylmethylphosphine]

RL: PREP (Preparation)
(preparation of)

IT 123-91-1P, p-Dioxane

RL: PREP (Preparation)

(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [ethylenebis(ethylphosphinidene)]dilithium (2:1))

AB cf. CA 58, 13414g. K mononocyclohexylphosphide (I) and KPHEt (II) react with (CH2Cl)2 to yield 1,2-ethylenebis(monocyclohexylphosphine) (III) and (EtPHCH2)2 (IV), resp., which with MeI or S yield the corresponding phosphonium salts or disulfides, resp. Compds. containing asym. P atoms could be separated in 2 cases into the racemic and meso forms. The presence of the P-H bond in III and IV is demonstrated by their IR spectra and by metalation. Di-Li phosphides of the type (RLiPCH2)2 react with (CH2Cl)2 to yield the corresponding 1,4-dialkyl-1,4-diphosphacyclohexanes, which with S give cis, trans isomeric IVa. Li dicyclo-hexylphosphide (V) (25 g.) in 300 cc. refluxing Et2O treated dropwise with stirring with 6.1 g. (CH2Cl)2 in 50 cc. Et2O gave 1.2 l. C2H4; the mixture cooled and filtered yielded 19 g. tetracyclohexyldiphosphine (VI), m. 169°. V (11 g.) in 80 cc. THF treated with cooling slowly with 2.7 g. (CH2Cl)2 at 0° gave 100 cc. C2H4 and 2 g. VI; the filtrate concentrated and diluted with 5 cc. EtOH yielded 6 g. 1,2-ethylenebis(dicyclohexylphosphine), m. 96-7°. Et2PLi (15.7 g.) from 13.8 g. PhLi and 16 g. Et2PH in 150 cc. refluxing Et2O treated dropwise with stirring with 8.1 g. (CH2Cl)2 gave 900 cc. C2H4; the mixture cooled, filtered, concentrated, and distilled

yielded 6 g. (Et2P)2 [characterized with EtI h in Et2O as (Et2PPEt3)I, m. 100°] and 5 g. (Et2PCH2)2 (VII) (characterized with 60% HI as VII.2HI, m. 181°). I (40 g.) in 400 cc. heptane treated with stirring at -20° with 12 g. (CH2Cl)2 in 50 cc. heptane, heated 2 h. at 70-5°, diluted with 100 cc. H2O, worked up, and the crude product chromatographed on AlcOa yielded 26 g. III, air-sensitive, b4 162-8°. II (20 g.) in 500 cc. Pr2O with 9 g. (CH2Cl)2 in 50 cc. Pr2O gave 5 g. EtPH2 and 6 g. IV, b18 90°. III (5.0 g.) and 6.0 g. MeI refluxed 5-10 min. in 50 cc. Et2O, refrigerated several days, filtered, the residue dissolved in 250 cc. hot EtOH, and cooled gave 4.1 g. III.2 MeI, m. 300-1°; the filtrate concentrated to 10-15 cc. yielded 1.9 g. low-melting III.2MeI, m. 147-50°. IV (2.0 g.) in 20 cc. Et2O and 4.0 g. MeI kept 4-5 h. at room temperature, the Et2O decanted, and the residue recrystd. from 60 cc. EtOH gave 3.8 g. IV.2MeI, m. 158-60°. III (12.5 g.) in 100 cc. C6H6 and 3.1 g. S kept several hrs., shaken with 50 cc. 2N NaOH, and the aqueous phase acidified with dilute H2SO4 yielded 1.9

g.

1,2-ethylenebis(cyclohexyldithiophosphinic acid); the C6H6 phase concentrated, and the residue fractionally recrystd. from Me2CO gave 4.9 g. dl1,2-ethylenebis(cyclohexylphosphine sulfide) (VIII), m. 155-8°,

and 1.9 g. the meso isomer, m. 125-30°. IV (2.0 g.) and 0.9 g. S in 20 cc. C6H6 yielded 1.8 g. [CH2P(S)HET]2, m. 109-10° (aqueous Me2CO). VIII (218.7 g.) treated with 5 cc. MeLi-Et2O containing 156.5 mg. MeLi gave 30 cc. CH4; a similar run with 212.8 mg. VIII yielded 29.5 cc. CH4; the reaction mixture filtered gave the solid di-Li derivative of VIII with 4 mol Et2O of crystallization III (6.0 g.) treated with cooling with 50 cc.

PhLi-Et2O

(73 mg. PhLi/cc.) gave 5.6 g. di-Li derivative (IX) of III. IV (2.0 g.) in 5 cc. dioxane and 22 cc. PhLi-Et2O (94 mg. PhLi/cc.) yielded 4 g. di-Li derivative (X) of IV. IX (8.1 g.) in 50 cc. refluxing Et2O treated dropwise with 3 g. (CH2Cl)2 in 20 cc. Et2O, refluxed 2 h., diluted with 10 cc. dioxane, filtered through kieselguhr, and distilled gave 1,4-dicyclohexyl-1,4-diphosphacyclohexane (XI), b2 225-30°. X (12.5 g.) in 30 cc. Et2O and 4.0 g. (CH2Cl)2 in 20 cc. Et2O gave 3.8 g. 1,4-di-Et analog (XII) of XI, light yellow, air-sensitive oil, b4 135-45°. XI (2.0 g.) and 0.45 g. S in 21 cc. C6H6 refluxed 10 min. and evaporated, and the residue crystallized from 15 cc. hot EtOH gave 0.8 g. IVA (R = cyclohexyl), m. 325 6°; the filtrate treated with 1 cc. H2O gave 0.18 g. low-melting form, m. 250 5° (C6H6). XII (2.0 g.) and 0.73 g. S in 20 cc. C6H6 evaporated gave similarly 0.1 g. IVA (R = Et), m. 225 35°. XI (2.0 g.) and 20 cc. 3% H2O2 shaken 2-3 h. and evaporated, and the residue resuspended in EtOH and filtered off gave 1.7 g. 1,4-dioxide of XI, m. 260-75°. PHCH2CH2PH (11I) R (S) (S) R (IVA)

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ACCESSION NUMBER: 1962:31535 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 56:31535

ORIGINAL REFERENCE NO.: 56:5996g-i,5997a-e

TITLE: Alkali metal-phosphorus compounds and their reactions.

XI. The reaction of potassium cyclohexylphosphide with dihaloalkanes

AUTHOR(S): Issleib, Kurt; Doell, Gerhard

CORPORATE SOURCE: Univ. Halle, Germany

SOURCE: Chemische Berichte (1961), 94, 2664-9

CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

TI Alkali metal-phosphorus compounds and their reactions. XI. The reaction of potassium cyclohexylphosphide with dihaloalkanes

IT Reactivity (of phosphorus compds. with alkali metals)

IT Alkylene halides (reaction with (cyclohexylphosphino)lithium)

IT Lithium, [hexamethylenebis(cyclohexylphosphinidene)]di-, compound with p-dioxane

Lithium, [tetramethylenebis(cyclohexylphosphinidene)]di-

RL: PREP (Preparation)

IT 65105-85-3 107712-89-0 108652-55-7

(Derived from data in the 7th Collective Formula Index (1962-1966))

IT 75-15-0, Carbon disulfide (compds. with tetramethylenebis[cyclohexylmethylphosphine])

IT 7723-14-0, Phosphorus (compds., alkali metal derivs. of organic)

IT 822-68-4P, Phosphine, cyclohexyl- 3040-71-9P, Tetraphosphetane, tetracyclohexyl- 3040-77-5P, Phosphine, cyclohexyl-, compound with HI

88591-62-2P, Phosphinodithioic acid, tetramethylenebis[cyclohexyl-

90050-09-2P, Phosphine, cyclohexylmethyl- 90114-83-3P, Phosphine,

tetramethylenebis[cyclohexyl- 90229-07-5P, Phosphinodithioic acid,

tetramethylenebis[cyclohexyl-, nickel salt 91725-33-6P, Phosphine,

cyclohexyl-, compound with HBr 92799-67-2P, Phosphine,

cyclohexyldimethyl-, compound with HI 94376-93-9P, Phosphinodithioic acid,

hexamethylenebis[cyclohexyl- 97340-94-8P, Lithium, [trimethylenebis(ethylphosphinidene)]di-, compound with p-dioxane 97472-13-4P, Phosphine, trimethylenebis[cyclohexyl- 98176-10-4P, Lithium, [pentamethylenebis(cyclohexylphosphinidene)]di- 98283-36-4P, Phosphine, trimethylenebis[cyclohexylmethyl-, compound with HI 98341-94-7P, Phosphine, pentamethylenebis[cyclohexyl- 98884-44-7P, Phosphine, tetramethylenebis[cyclohexylmethyl-, compound with HI 99080-60-1P, Phosphine, pentamethylenebis[cyclohexylmethyl-, compound with HI 99729-90-5P, Phosphine, hexamethylenebis[cyclohexylmethyl-, compound with HI 99812-62-1P, Phosphine, tetramethylenebis[cyclohexylmethyl-, compound with CS2 99926-44-0P, Phosphine, hexamethylenebis[cyclohexyl- 101016-80-2P, Phosphinic acid, hexamethylenebis[cyclohexyl- 108271-78-9P, Lithium, [trimethylenebis(cyclohexylphosphinidene)]di-, compound with p-dioxane
 RL: PREP (Preparation)
 (preparation of)

IT 123-91-1P, p-Dioxane
 RL: PREP (Preparation)
 (reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [hexamethylenebis(cyclohexylphosphinidene)]dilithium)

IT 123-91-1P, p-Dioxane
 RL: PREP (Preparation)
 (reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [trimethylenebis(cyclohexylphosphinidene)]dilithium)

IT 123-91-1P, p-Dioxane
 RL: PREP (Preparation)
 (reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [trimethylenebis(phenylphosphinidene)]dilithium)

IT 93386-85-7, Potassium, (cyclohexylphosphino)-
 (reaction with alkylene halides)

IT 28240-66-6, Phosphine, trimethylenebis[phenyl-
 (P,P')-dilithium derivative, compound with p-dioxane)

AB K cyclohexylphosphide (I) with 1,3-, 1,4-, 1,5-, and 1,6-dihaloalkanes yields the corresponding C8H11PH(CH2)nPHC6H11 (n = 3-6) (II). I with (CH2Br)2 or CH2Cl2 yields in addition to C2H4, cyclohexylphosphine (III), and tetracyclohexylcyclotetraphosphine (IV), because of a metal-halogen exchange, also methylcyclohexylphosphine (V) and IV. The 2 H atoms of the II are replaceable by Li to yield the corresponding di-Li derivs. (VI). The preparation of phosphonium salts and dithio acids of the types [Me(C6H11)HP(CH2)nPH(C6H11)Me]I2 (VIA) and C6H11(HS)(S)P(CH2)nP(S)(SH)C6H11 (n = 3-6) (VII), resp. The reaction of III with (CH2Br)2 yields by halogen-H exchange IV, III.HBr, and C2H4. III (35 g.), 500 cc. heptane, and 7.5 g. K refluxed 4-6 hrs. with stirring, cooled, and filtered gave 39 g. I, air- and moisture-sensitive solid. I in 300 cc. refluxing C6H6 treated during 1 hr. with an appropriate dihaloalkane in 100 cc. C6H6, refluxed 0.5 hr., and decomposed with 50 cc. O-free H2O, and the C6H6 layer worked up gave the corresponding II. I (20 g.) and 9.5 g. Cl(CH2)6Cl gave 13.5 g. II (n = 6) (VIII), b2 200-10°, m. 13-14°. I (20 g.) and 14 g. Br(CH2)5Br yielded 11.8 g. II (n = 5) (IX), b2 177°. I (20 g.) and 8 g. Cl(CH2)4Cl gave 11.7 g. II (n = 4) (X), b2 165-6°, m. 4°. I (20 g.) and 12.5 g. Br(CH2)3Br yielded 10.5 g. II (n = 3) (XI), b2 151°. The appropriate II in 25 cc. Et2O treated with excess MeI, kept several hrs. and filtered gave the corresponding VIA (n, m.p., g. yield, g. amount and II used are given): 6, 158-60°, 2.9, 2, VIII; 5, 160-2°, 2.3, 2, IX; 4 (XII), 185-70°, 2.7, 2, X; 3, 133-50°, 2.3, 2, XI. The appropriate II in 25-30 cc. Et2O treated dropwise with the calculated amount of PhLi in Et2O and filtered without or with previous addition of dioxane gave the following di-Li derivs. (n, number of moles of complexed dioxane, g., amount II, and cc. volume dioxane used and g., yield of product are given): 6, 2, 2, 3, 3.1; 5, 0, 2, 0, 2; 4, 0, 2, 0, 2; 3, 2, 2, 3,

3.1. VIII (2 g.) in 25 cc. C6H6 treated with 0.8 g. S, boiled briefly, and evaporated, and the oily residue dissolved in aqueous NaOH, filtered, and acidified gave 1.6 g. VII (n = 6), m. 108-10° (aqueous MeOH). X (5 g.) and 2.2 g. S gave similarly 6.1 g. VII (n = 4) (XIII), m. 156-7°. VIII (2 g.) in 50 cc. C6H6 treated with a stream of air until evaporated gave 1.9 g. 1,6-hexamethylenebis(cyclohexylphosphinic acid), m. 140-2° (PhMe). XII (1 g.), 10 cc. H2O, 20 cc. Et2O, and 0.5 g. NaOH shaken briefly, and the Et2O layer dried and treated with 1-2 cc. CS2 gave 0.59 g. pale red 1,4-butylenebis(methylcyclohexylphosphine)-CS2, adduct, which decomposed gradually. XIII (1.5 g.), 0.5 g. NiBr2, and 25 cc. C6H6 refluxed 5 hrs. and filtered yielded 1 g. Ni salt of XIII, decomposed 355°. III (10 g.) and 8 g. (CH2Br)2 refluxed 2 hrs., triturated with hot EtOH, and recrystd. from C6H6 gave 1.6 g. IV, m. 220°; 280 cc. C2H4 was evolved during the reaction; III.HBr, m. 143-5°, had sublimed during the reaction into the condenser; it decomposed in EtOH or in air into III and HBr. I (10 g.) in 250 cc. hot C6H6 treated dropwise with stirring with 6 g. (CH2Br)2 in 50 cc. C5H6 gave 610 cc. C2H4; the mixture treated with 20 cc. O-free H2O, the C6H6 layer evaporated, and the residue diluted with 100 cc. Et2O gave 2.9 g. IV, leaflets, m. 220°; the Et2O filtrate concentrated to 10-20 cc. and treated with HI gave III.HI, leaflets,

m.

170° (EtOH). I (10 g.) and 2.7 g. CH2Cl2 in 250 cc. C6H6 gave similarly 1.7 g. IV, m. 220°; the Et2O layer gave 2 g. V, b3 30-1°. V (1 g.) and 2 g. MeI in 15 cc. Et2O yielded 1 g. dimethylcyclohexylphosphonium iodide, m. 140-2° (EtOH). I (10 g.) in 250 cc. C6H6 treated dropwise with stirring with 5.2 g. Br in 50 cc. C6H6, filtered, concentrated, and diluted with Et2O yielded 2.6 g. IV, m. 220°; the filtrate concentrated and treated with HI gave III.HI, m. 170°.

L6 ANSWER 16 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1962:31534 CAPLUS <LOGINID:20080918>

DOCUMENT NUMBER: 56:31534

ORIGINAL REFERENCE NO.: 56:5995f-4,5996a-g

TITLE: Alkali metal-phosphorus compounds and their reactions.

X. Alkali phosphides of the type Ar(Li)P(CH2)nP(Li)Ar

and their reaction with alkyl and cycloalkyl halides

AUTHOR(S): Issleib, Kurt; Krech, Frieder

CORPORATE SOURCE: Univ. Halle, Germany

SOURCE: Chemische Berichte (1961), 94, 2656-63

CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 56:31534

TI Alkali metal-phosphorus compounds and their reactions. X. Alkali phosphides of the type Ar(Li)P(CH2)nP(Li)Ar and their reaction with alkyl and cycloalkyl halides

IT Reactivity

Reactivity

(of phosphorus compds. with alkali metals)

IT Alkylene halides

(reaction with (cyclohexylphosphino)lithium)

IT Alkyl halides

(reaction with phosphine derivs.)

IT Lithium, [pentamethylenebis(phenylphosphinidene)]di-, compound with p-dioxane

RL: PREP (Preparation)

IT 100321-06-0 107712-89-0 108486-25-5 108652-55-7

(Derived from data in the 7th Collective Formula Index (1962-1966))

IT 7723-14-0, Phosphorus

(compds., alkali metal derivs. of organic)

- IT 3302-87-2P, Phospholane, 1-phenyl- 6050-22-2P, 1,2-Diphospholane, 1,2-diphenyl- 25900-28-1P, Phosphine sulfide, trimethylenebis[ethylphenyl- 25900-30-5P, Phosphine sulfide, tetramethylenebis[cyclohexylphenyl- 25900-32-7P, Phosphine sulfide, pentamethylenebis[ethylphenyl- 28240-67-7P, Phosphine, tetramethylenebis[phenyl- 65105-85-3P, 1,2-Diphospholane, 1,2-diphenyl-, 1,2-disulfide 72144-83-3P, Phosphine, tetramethylenebis[cyclohexylphenyl- 73191-51-2P, Phosphine, hexamethylenebis[phenyl- 83152-25-4P, Phosphine, hexamethylenebis[ethylphenyl- 89399-68-8P, Phosphine sulfide, pentamethylenebis[cyclohexylphenyl- 90116-42-0P, Phosphine, pentamethylenebis[cyclohexylphenyl- 97340-94-8P, Lithium, [trimethylenebis(ethylphosphinidene)]di-, compound with p-dioxane 98170-74-2P, Phosphine, hexamethylenebis[cyclohexylphenyl- 98341-81-2P, Phosphine, pentamethylenebis[phenyl- 99888-09-2P, Phosphine, trimethylenebis[ethylphenyl- 100151-95-9P, Phosphine, tetramethylenebis[ethylphenyl- 100151-96-0P, Phosphine sulfide, tetramethylenebis[ethylphenyl- 100170-44-3P, Phosphine oxide, tetramethylenebis[ethylphenyl- 100335-42-0P, Nickel, dibromo[pentamethylenebis[ethylphenylphosphine]]- 100407-14-5P, Phosphine, pentamethylenebis[ethylphenyl- 100433-15-6P, Phosphine, pentamethylenebis[(p-ethylphenyl)-, nickel complex 100776-08-7P, Lithium, [hexamethylenebis(phenylphosphinidene)]di-, compound with p-dioxane 100997-04-4P, Phosphine sulfide, hexamethylenebis[ethylphenyl-, stereoisomers 101320-02-9P, Phosphonium, tetramethylenebis[diethylphenyl-iodide] 106524-86-1P, Phosphine, pentamethylenebis[phenyl-, compound with HI 106572-33-2P, Phosphine sulfide, hexamethylenebis[cyclohexylphenyl- 107101-27-9P, Phosphine oxide, tetramethylenebis[cyclohexylphenyl- 108271-53-0P, Lithium, [tetramethylenebis(phenylphosphinidene)]di-, compound with p-dioxane
- RL: PREP (Preparation)
(preparation of)
- IT 123-91-1P, p-Dioxane
- RL: PREP (Preparation)
(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [hexamethylenebis(phenylphosphinidene)]dilithium)
- IT 123-91-1P, p-Dioxane
- RL: PREP (Preparation)
(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [tetramethylenebis(phenylphosphinidene)]dilithium)
- IT 28240-66-6, Phosphine, trimethylenebis[phenyl- 28240-67-7, Phosphine, tetramethylenebis[phenyl- 73191-51-2, Phosphine, hexamethylenebis[phenyl- (P,P'-dilithium derivative, compound with p-dioxane)
- IT 98341-81-2, Phosphine, pentamethylenebis[phenyl- (P,P'-dilithium derivative, compds. with p-di-oxane)
- IT 822-68-4, Phosphine, cyclohexyl- (P-potassium derivative, reaction with alkylene halides)
- AB cf. CA 55, 15428a.-Disecundary phosphines of the general formula ArHP(CH₂)_nPHAr (n = 3-6) with PhLi yield the corresponding di-Li phosphides which with alkyl and cycloalkyl halides give unsym. substituted phosphines. Ph(Li)P(CH₂)₃P(Li)Ph (I) yields with EtLi, in addition to [EtPhPCH₂]₂CH₂ (II), because of a metal-halogen exchange 1,2-diphenyl-1,2-diphosphacyclopentane (III) which can also be obtained from I and (CH₂Br)₂. The unsym. ditertiary phosphines can be converted to the corresponding disulfides, bisphosphonium salts, or dioxides by reaction with S, alkyl halides, or by oxidation, resp. NaPPh (IV) (50 g.) in 200 cc. Et₂O treated dropwise with stirring with 43.5 g. Br(CH₂)₅Br, refluxed 0.5 hr., cooled, diluted with 150 cc. H₂O, and worked up gave 43.5 g. air-sensitive [PhPH(CH₂)₂]₂CH₂ (V), b₄-5 211-14°. V (6 g.) in 150 cc. Et₂O saturated with dry HI and filtered gave 4.1 g. V.HI, m. 155-7° (repptd. from EtOH with Et₂O). IV (48.5 g.) treated with 28.5 g. Cl(CH₂)₆Cl in 170 cc. Et₂O yielded 48 g.

[PhHP(CH₂)₃]₂ (VI), b₄ 207-11°. IV (56.2 g.) in 200 cc. Et₂O and 27 g. Cl(CH₂)₄Cl gave similarly 12.5 g. 1-phenylcyclohexylmethylenephosphine, b₄ 110-12°, 29.6 g. [PhHP(CH₂)₂]₂, b₄ 195-7°, and 5.6 g. PhPH₂. VI (1.65 g.) in 15 cc. Et₂O treated dropwise with 15 cc. PhLi solution containing 61 mg./cc., kept several hrs., added with stirring to 10

cc.

dioxane, and filtered yielded 2.3 g. [Ph(Li)P(CH₂)₃]₂.2C₄H₈O₂ (VII). V (13.4 g.) in 150 cc. Et₂O with 85 cc. PhLi-Et₂O containing 92 mg./cc. and 30 cc. dioxane refluxed 0.5 hr. yielded similarly 27.3 g. [Ph(Li)P(CH₂)₂]₂.2C₄H₈O₂ (VIII). V (0.8 g.) in 20 cc. Et₂O, 50 cc. PhLi-Et₂O (93.3 mg./cc.), and 5 cc. dioxane gave 1.2 g. [Ph(Li)P(CH₂)₂]₂.2C₄H₈O₂. VIII heated 5 hrs. at 100° gave [Ph(Li)P(CH₂)₂]₂.2C₄H₈O₂. [PhHP(CH₂)₂]₂ (2.1 g.) in 120 cc. Et₂O with 14 cc. PhLi-Et₂O (92 mg./cc.) and 10 cc. dioxane gave 2.8 g. [Ph(Li)P(CH₂)₂]₂.2C₄H₈O₂ (VIIIA). VII (12.5 g.) in 200 cc. Et₂O treated dropwise with 12.4 g. EtI in 50 cc. Et₂O, refluxed 0.5 hr., and diluted with 100 cc. H₂O, and the Et₂O layer worked up gave 8.3 g. [EtPhP(CH₂)₃]₂ (IX), needles, m. 40-1°, b_{0.1} 193-6°. IX (2.84 g.) in 20 cc. C₆H₆ refluxed briefly with 0.51 g. S, the C₆H₆ distilled, and the residue treated 24 times with 10-cc. portions MeOH left 1.04 g. (crude) meso-disulfide of IX, needles, m. 163-4° (Me₂CO); the MeOH extract evaporated gave 1.65 g. racemic disulfide of IX, m. 88-90°. VII (26 g.) in 200 cc. PhMe treated with stirring with 17.3 g. cyclohexyl bromide in 20 cc. PhMe, boiled briefly, filtered, and evaporated gave 20.6 g. 1,6-hexamethylenebis(cyclohexylphenylphosphine) (X), viscous oil. X (2 g.) in 50 cc. C₆H₆ treated with 0.3 g. S gave 0.3 g. disulfide of X, m. 211-14° (C₆H₆). VIII (16.7 g.) and 17.3 g. EtI in 225 cc. Et₂O treated with 10 cc. dioxane, filtered, and distilled yielded 12 g. [EtPhP(CH₂)₂]₂.2CH₂ (XI), b₃ 211-13°. XI (2 g.), 0.4 g. S, and 20 cc. C₆H₆ gave 0.5 g. disulfide of XI, m. 115-16° (MeOH). XI (2 g.), 3 g. NIBr₂, and 35 cc. PhMe refluxed 2 hrs. yielded 2.1 g. dibromo-1,5-pentamethylenebis(ethylphenylphosphine)nickel, red-brown, m. 148-53° (PhMe). VIII (25.5 g.), 13.5 g. cyclohexyl bromide, and 230 cc. PhMe gave similarly an oily product which diluted with 150 cc. hot EtOH, filtered, and cooled yielded 14.8 g. 1,5-pentamethylenebis(cyclohexylphenylphosphine) (XII), oil at room temperature

XII

(3.02 g.), 0.43 g. S, and 35 cc. C₆H₆ yielded in the usual manner 0.43 g. disulfide of XII, needles, m. 184-5° (Me₂CO). VIIIA (30.9 g.), 33.7 g. EtI, and 300 cc. Et₂O gave in the usual manner 22.4 g. [EtPhP(CH₂)₂]₂ (XIII), b₄ 225-30°. XIII (2.7 g.), 0.6 g. S, and 50 cc. C₆H₆ gave 0.5 g. disulfide of XIII, needles, m. 179-80° (Me₂CO). XIII (2 g.) in 20 cc. Me₂CO treated with KMnO₄-Me₂CO until the color persisted, decolorized with a few drops 3% aqueous H₂O₂, filtered, and evaporated gave 0.25 g. [EtPhP(O)CH₂CH₂]₂, m. 159-61° (PhMe). XIII (2 g.) and 3 g. EtI in 40 cc. EtOH refluxed 0.5 hr. and refrigerated gave 2.9 g. [(Et₂PhPCH₂CH₂)₂]₂, m. 210-12° (EtOH). VIIIA (12.3 g.) and 6.3 g. cyclohexyl chloride in 170 cc. dioxane gave similarly 7 g. 1,4-tetramethylenebis(cyclohexylphenylphosphine) (XIV), m. 104-5°. XIV (1.5 g.), 0.3 g. S, and 50 cc. C₆H₆ yielded 0.36 g. disulfide of XIV, m. 248-50°. XIV (4.9 g.) oxidized in the usual manner with KMnO₄ gave 0.76 g. 1,4-tetramethylenebis(cyclohexylphenylphosphine oxide), m. 201-2°. I (11.5 g.) in 200 cc. Et₂O treated dropwise with EtCl in Et₂O, shaken immediately with two 100-cc. portions H₂O, and worked up gave 9.2 g. II, b₃ 196-200°. II(2.13 g.), 0.43 g. S, and 30 cc. C₆H₆ gave 0.56 g. disulfide of II, m. 139-40°. I.2C₄H₈O₂ (24.6 g.) in 200 cc. C₆H₆ and 20 cc. tetrahydrofuran treated during 3 hrs. with 10.3 g. (CH₂Br)₂ in 100 cc. C₆H₆ gave about 1110 cc. C₂H₄; the mixture filtered and evaporated gave 8.8 g. 1,2-diphenyl-1,2-diphosphacyclopentane (XV), b₄ 184-90°. XV (2.83 g.) and 0.7 g. S in 15 cc. C₆H₆ refluxed a few min. and evaporated yielded 1.74 g. 1,2-diphenyl-1,2-diphosphacyclopentane

1,2-disulfide, m. 178-80° (Me2CO).

L6 ANSWER 17 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1962:31533 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 56:31533

ORIGINAL REFERENCE NO.: 56:5995e-f

TITLE: Reaction of phosphine and of phosphorous acid with aldehydes. V. Reaction of hypophosphorous acid with formaldehyde and benzaldehyde

AUTHOR(S): Horak, J.; Ettel, V.

CORPORATE SOURCE: Inst. Chem. Technol., Prague

SOURCE: Collection of Czechoslovak Chemical Communications (1961), 26, 2410-17

CODEN: CCCCAC; ISSN: 0010-0765

DOCUMENT TYPE: Journal

LANGUAGE: German

TI Reaction of phosphine and of phosphorous acid with aldehydes. V.

IT Reaction of hypophosphorous acid with formaldehyde and benzaldehyde

IT Aldehydes

(reactions of, with P compds.)

IT 100321-06-0 108486-25-5

(Derived from data in the 7th Collective Formula Index (1962-1966))

IT 90-09-5P, Phosphinic acid, bis(α -hydroxybenzyl)- 2074-67-1P, Phosphinic acid, bis(hydroxymethyl)- 52705-43-8P, Phosphonous acid, (α -hydroxybenzyl)- 60672-77-7P, Phosphonous acid, (hydroxymethyl)-

RL: PREP (Preparation)

(preparation of)

IT 7803-51-2, Phosphine 13598-36-2, Phosphorous acid

(reaction with aldehydes)

IT 7803-51-2, Phosphine

(reactions of derivs. of, with aldehydes)

IT 50-00-0, Formaldehyde

(reactions of, with hypophosphorous acid)

IT 100-52-7, Benzaldehyde

(reactions of, with hypophosphorous acid, kinetics of)

IT 6303-21-5, Hypophosphorous acid

(reactions with HCHO or BzH, kinetics of)

AB The products of the reactions mentioned in the title have the structures:

RCHOHPO2H2 (I), RCHOHPO(OH)CHOHR (II), where R stands for H or Ph. The reaction rate is linearly proportional to the aldehyde and undissocd. H3PO2 concns. For the sake of simplicity it is possible to express the rate equations in the following way: $v = k [\text{CH}_2\text{O}]^{0.7} [\text{H}_3\text{PO}_2]^{(1-\alpha)}$ and $v = k [\text{PhCHO}]^{0.85} [\text{H}_3\text{PO}_2]^{(1-\alpha)}$, where α means the degree of the dissociation of H3PO2. The values of the velocity consts. at 90° are 0.295 ± 0.023 l.0.7 mole-0.7 hr.-1 (CH2O) and 0.136 ± 0.011 l.0.85 mole-0.85 hr.-1 (PhCHO). A reaction scheme is suggested.

L6 ANSWER 18 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1959:121944 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 53:121944

ORIGINAL REFERENCE NO.: 53:21843i,21844a-i

TITLE: Synthesis of 2,3:6,7-dibenzodiphenylene, and reaction of 1-bromo-2-iodonaphthalene with magnesium

AUTHOR(S): Ward, E. R.; Pearson, B. D.

CORPORATE SOURCE: Leicester Coll. Technol. Commerce, UK

SOURCE: Journal of the Chemical Society (1959) 1676-80

CODEN: JCSOA9; ISSN: 0368-1769

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 53:121944

TI Synthesis of 2,3:6,7-dibenzodiphenylene, and reaction of
1-bromo-2-iodonaphthalene with magnesium
IT 811-20-1 108632-57-1 126329-92-8 128633-89-6
129068-11-7

(Derived from data in the 6th Collective Formula Index (1957-1961))
IT 242-50-2P, 6H-Dibenzo[b,h]carbazole 252-44-8P, Dibenzo[a,g]biphenylene
258-47-9P, Dibenzo[b,h]biphenylene 13115-28-1P, 2-Naphthylamine,
3-nitro- 29492-74-8P, Acetamide, N-(5,6,7,8-tetrahydro-3-nitro-2-
naphthyl)- 50537-06-9P, Naphthalene, 1,2,3,4-tetrahydro-6-iodo-7-nitro-
102153-44-6P, Naphthalene, 2-bromo-3-iodo- 102153-71-9P, Naphthalene,
2-iodo-3-nitro- 102317-09-9P, 2,2'-Binaphthyl, 5,5',6,6',7,7',8,8'-
octahydro-3,3'-dinitro- 110939-85-0P, Dibenzo[b,h]biphenylene,
tetrahydro- 112376-54-2P, 1,2,3,4,8,9,10,11-Octahydrodinaphth[2,3-
b:2',3'-d]iodolium iodide 114164-69-1P, 3,3'-Bi[2-naphthylamine],
5,5',6,6',7,7',8,8'-octahydro-
RL: PREP (Preparation)

(preparation of)
IT 7439-95-4, Magnesium
(reaction with 1-bromo-2-iodonaphthalene)

IT 90948-03-1, Naphthalene, 1-bromo-2-iodo-
(reaction with Mg)

AB 2,3:6,7-Dibenzodiphenylene (I) was synthesized by the pyrolysis of
5,6,7,8,5',6',7',8'-octahydro-2,2'-dinaphthyl-3,3'-iodonium iodide (II)
and was accompanied by 2 other products which appear to be hydrogenated
derivs. of I. The pyrolysis of the corresponding 3,3'-tetrazonium sulfate
(III) was also studied. 1-Bromo-2-iodonaphthalene (IV) was prepared as
follows. 1-Bromo-2-naphthylamine (10 g.) in 7 cc. warm H₂SO₄ and 20 cc.
H₂O diazotized at 0° by rapid addition of 5 g. NaNO₂ in 15 cc. H₂O,
the solution added to 6 g. iodine and 18 g. KI in 120 cc. H₂O, stirred 1 hr.,
the solids collected, shaken with 200 cc. 10% aqueous Na₂S₂O₃, collected
again, washed, dried, dissolved in C₆H₆, and passed through Al₂O₃ gave 6 g.
IV, m. 94°. 3-Bromo-2-naphthylamine (10 g.) in 4 g. NaNO₂ in 40
cc. H₂SO₄ stirred 1 hr. below 30° into 80 cc. AcOH, and the solution
treated with iodine-KI gave 10 g. 2-bromo-3-iodonaphthalene, m.
120° (alc.-EtOAc). 3-Nitro-2-naphthylamine (10 g.) in 120 cc. AcOH
similarly gave 14 g. 2-iodo-3-nitronaphthalene, m. 89° (alc.).
6-Acetamido-1,2,3,4-tetrahydronaphthalene (50 g.) in 810 cc. Ac₂O treated
dropwise below 25° with 16.7 cc. Ac₂O and 16.7 cc. HNO₃, left
overnight, poured into ice-H₂O, and the liquid siphoned off gave 35-9%
6-acetamido-1,2,3,4-tetrahydro-7-nitronaphthalene (V). Further
amts. were obtained by evaporation of the mother liquor to dryness and
separation

from the 5-nitro isomer by chromatography on Al₂O₃ in C₆H₆-EtOAc.
Diazotizing V and adding the diazonium solution to aqueous iodine-KI
underlaid with CHCl₃ gave 60-80% 1,2,3,4-tetrahydro-6-iodo-7-
nitronaphthalene (VI). VI (1 g.) heated during 0.5 hr. to 132°
with portionwise addition of 0.6 g. Cu-bronze, the temperature kept below
140°, the heating continued another 0.5 hr., extracted with hot C₆H₆,
and chromatographed on Al₂O₃ gave 0.42 g. 3,3'-dinitro-5,5',6,6',7,7',8,8'-
octahydro-2,2'-binaphthyl (VII), m. 190-1°. VII (3.5 g.) in 165
cc. EtOAc and 15 cc. alc. in the presence of 3 g. Raney Ni and
treated with H at 75° and atmospheric pressure for 8 hrs. gave 2.2 g.
3,3'-diamino-5,5',6,6',7,7',8,8'-octahydro-2,2'-binaphthyl (VIII), m.
193-4° (EtOAc-alc.). Reduction at room temperature with N₂H₄ in the
presence of Raney Ni or Pd-C was not successful. VIII (0.15 g.)
in 1.5 cc. HCl and 2 cc. H₂O was treated at 0° with 0.2 g. NaNO₂ in
1 cc. H₂O, the diazonium solution decomposed by addition to 0.3 g. iodine and

1 g. KI in 20 cc. H₂O, after 1 hr. the solids collected, dried, and extracted with
C₆H₆ to give 92% II, decompose 240-5°. The C₆H₆ extract washed with aqueous
Na₂S₂O₃, dried, and chromatographed on Al₂O₃ gave 0.016 g. green crystals,

m. 208°. An intimate mixture of 0.3 g. II and 3 g. cuprous oxide under N at 0.3 mm. heated 0.5 hr. at 340°, the temperature kept 1 hr. at 220°, and the product extracted 8 hrs. in a Soxhlet apparatus gave yellow plates. The C6H6 mother liquor was chromatographed on Al2O3 giving a yellow solid. When freshly prepared Cu2O was used, 0.288 g. II afforded very thin plates of I, sublimed at 344-6° without melting. The 3 mg. yellow needles m. 224-6°. With an old sample of Cu2O, 0.3 g. II gave 4 mg. of product, m. 300-2°, considered to be a tetrahydrodibenzodiphenylene and 4 mg. of needles, m. 224-6°. The appropriate diamine (0.5 g.) in a solution of 0.4 g. NaNO2 and 4 cc. H2SO4 was added below 30° to 9 cc. AcOH, after stirring 1 hr. 100 cc. cold Et2O added, the mixture left 1 hr. at room temperature, the III collected, washed, dried, ground with 7 g. Cu2O, covered with 5 g. more Cu2O, and the whole pyrolyzed at dull red heat and 0.3 mm., cooled in vacuo, extracted with C6H6, and concentrated to give 5 mg. of a dibenzocarbazole, m. 299-301°. Chromatography of the mother liquor gave 36 mg. of an octahydrodibenzocarbazole, m. 157-8° (alc.), and the residue of 3 mg. was not further investigated. IV (11 g.) in 120 cc. Et2O added during 0.5 hr. to a mixture of 1.75 g. Mg and 50 cc. Et2O, the reaction being initiated by addition of iodine and a trace of MeMgI, and conducted under N. After refluxing 1 hr. 60 cc. dry C6H6 was added at 0° and the refluxing continued 1 hr. Unchanged Mg was removed and carboxylation carried out by pouring on to a slurry of Et2O and solid CO2 followed by acidification with 500 cc. 20% HCl; fractional crystallization gave 0.5 g. 2,2'-binaphthyl, 12 mg. of orange plates, m. 271° (red trinitrofluorenone derivative of m. 267°), and a heavy viscous liquid which could not be distilled at 3 mm.

=> log h		
COST IN U.S. DOLLARS	SINCE FILE	TOTAL
	ENTRY	SESSION
FULL ESTIMATED COST	95.86	111.98
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE	TOTAL
	ENTRY	SESSION
CA SUBSCRIBER PRICE	-14.40	-14.40

SESSION WILL BE HELD FOR 120 MINUTES
STN INTERNATIONAL SESSION SUSPENDED AT 10:42:02 ON 18 SEP 2008

Connecting via Winsock to STN

Welcome to STN International! Enter x:x

LOGINID:sssptal745sxt

PASSWORD:

* * * * * RECONNECTED TO STN INTERNATIONAL * * * * *
SESSION RESUMED IN FILE 'CAPLUS' AT 10:46:24 ON 18 SEP 2008
FILE 'CAPLUS' ENTERED AT 10:46:24 ON 18 SEP 2008
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COST IN U.S. DOLLARS	SINCE FILE	TOTAL
	ENTRY	SESSION
FULL ESTIMATED COST	95.86	111.98
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE	TOTAL

CA SUBSCRIBER PRICE

ENTRY SESSION
-14.40 -14.40

=> d his

(FILE 'HOME' ENTERED AT 10:40:00 ON 18 SEP 2008)

FILE 'REGISTRY' ENTERED AT 10:40:21 ON 18 SEP 2008

L1 80 S (2-7)/LI AND P AND 3.5-8/O

FILE 'CAPLUS' ENTERED AT 10:40:52 ON 18 SEP 2008

L2 59 S L1

L3 8 S L2 AND (TI OR V OR CR OR MN)

L4 11 S L2 AND (FE OR CO OR NI OR CU OR ZR)

L5 6 S L2 AND (NB OR MO OR RU OR AG OR TA OR W OR PT OR AU)

L6 18 S L3 OR L4 OR L5

=> s l1

L7 59 L1

=> s l7 not nitride

276980 NITRIDE

34076 NITRIDES

285950 NITRIDE

(NITRIDE OR NITRIDES)

L8 55 L7 NOT NITRIDE

=> s l8 and (electrolyte or ion?)

278071 ELECTROLYTE

143681 ELECTROLYTES

333082 ELECTROLYTE

(ELECTROLYTE OR ELECTROLYTES)

2212647 ION?

L9 1 L8 AND (ELECTROLYTE OR ION?)

=> d

L9 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2008 ACS on STN

AN 2006:558613 CAPLUS <<LOGINID::20080918>>

DN 145:66266

TI Nonaqueous electrolyte secondary batteries having lithium phosphorus fluoride oxide or sodium/potassium hydrogen fluoride on surface or interface of cathode active mass particles

IN Kamibo, Yasushi

PA GS Yuasa Corp., Japan

SO Jpn. Kokai Tokkyo Koho, 15 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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PI	JP 2006156008	A	20060615	JP 2004-342240	20041126
PRAI	JP 2004-342240		20041126		

=> d 1 ibib ti it abs

L9 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2006:558613 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 145:66266

TITLE: Nonaqueous electrolyte secondary batteries having lithium phosphorus fluoride oxide or sodium/potassium hydrogen fluoride on surface or interface of cathode active mass particles

INVENTOR(S): Kamibo, Yasushi

PATENT ASSIGNEE(S): GS Yuasa Corp., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 15 pp.
CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2006156008	A	20060615	JP 2004-342240	20041126

PRIORITY APPLN. INFO.: JP 2004-342240 20041126

TI Nonaqueous electrolyte secondary batteries having lithium phosphorus fluoride oxide or sodium/potassium hydrogen fluoride on surface or interface of cathode active mass particles

IT Secondary batteries
(lithium; nonaq. electrolyte secondary batteries having lithium phosphorus fluoride oxide or sodium/potassium hydrogen fluoride on surface or interface of cathode active mass particles)

IT Battery cathodes

Safety
(nonaq. electrolyte secondary batteries having lithium phosphorus fluoride oxide or sodium/potassium hydrogen fluoride on surface or interface of cathode active mass particles)

IT 1333-83-1, Sodium fluoride (Na(HF2)) 7789-29-9, Potassium hydrogen fluoride (KHF2) 15772-79-9 845910-47-6, Lithium phosphorus fluoride oxide (LiPF2O2) 890852-51-4, Lithium phosphorus fluoride oxide (Li2PF3O2) 890852-52-5, Lithium fluoride oxide phosphide (LiO-4F0-6O0-4P)

RL: DEV (Device component use); USES (Uses)
(nonaq. electrolyte secondary batteries having lithium phosphorus fluoride oxide or sodium/potassium hydrogen fluoride on surface or interface of cathode active mass particles)

AB The batteries have, on surface and/or interface of cathode active mass particles, compds. containing alkali metals, F, and P, O, and/or H. The compds. prevent oxidative decomposition of electrolyte solvents on the surface of the active mass particles and improve safety of the batteries, especially when excess charging at high temperature

=> log h